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Journal

—OF THE—

American Ceramic Society

A Monthly Journal devoted to the Arts and Sciences Related to the Silicate Industries.

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JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 2 **July, 1919** **No. 7**

EDITORIALS.

CERAMIC TERMS AND DEFINITIONS.

In the ceramic industry there has been some confusion from time to time as to the exact meaning of certain terms and in other cases it has been the practice to use several words interchangeably to indicate the same product or the same process.

Now that ceramic engineering and ceramic literature have become well established, it would seem advisable to simplify and clear up some of the terms and definitions in use.

First, there are the words "firing" and "burning" which are used interchangeably. Which is more correct, or which more desirable? Ordinarily, "burning" implies consuming by fire, reducing to ashes, injury by fire or heat (Webster). A cook may burn the toast or the cake, a child may burn its fingers, or a lamp burn a hole in a window curtain. "Burn" in all of these sentences implies either injury or destruction. It is true that Webster states that "burning" may indicate perfecting or improving by fire and heat, but this is not the popular conception of the term. In a chemical sense "burning" denotes oxidation and this again complete change of the original substance. On the other hand, to "fire" or the act of firing, means, according to Webster, to set on fire, to kindle, to ignite, or *to subject to intense heat.* It would seem, therefore, that of the primary meanings of the two words, as recognized by such authorities as Webster, the term "firing" would be more correct than "burning" as applied to the finishing of ceramic objects in the kiln. As these terms are generally or popularly understood, the term "firing" would provide a more satisfactory conception of the operation than "burning."

Of the terms "firing" and burning," therefore, the former seems more suitable and should be employed to the exclusion of the latter term.

We also desire to call attention to the words "slip" and "engobe." Webster defines the word "slip" as any covering easily slipped on (slip covers for chairs, underwaist, often of contrasting color, etc.). In this sense the word "slip" may very properly be applied to clay slips which are readily applied to surfaces of ceramic wares to provide a particular color or finish different from that of the underlying body or base. The word "engobe" is of French origin and is defined by Webster as a white or colored *slip* applied as a thin coating to earthenware, often as a support for a second glaze or enamel. If an engobe then is merely a slip, why not stick to the original term, particularly since the word slip in the ceramic sense has identically the same fundamental meaning as the term slip applied to furniture covers, wearing apparel, etc.? All evidence indicates that the term slip is entirely correct and consistent and this word should, therefore, be used in ceramic literature to the exclusion of the word "engobe."

These cases illustrate the need of adjustment in ceramic nomenclature. Other cases will no doubt come to the minds of our readers, as the frequent but incorrect confusion of the terms "fire clays" and "refractory clays," kilns and furnaces, porcelain and china, clay and kaolin, etc.

In the printing of THIS JOURNAL and in the preparation of an index for the nineteen volumes of the Transactions of the American Ceramic Society, it will be the purpose of the Committee on Publications to bring about as far as possible the elimination of superfluous or apparently incorrect expressions or terms and the coöperation of the membership of the American Ceramic Society and of the readers of this JOURNAL will be appreciated in this work.

PATRONIZING HOME MARKETS

Although the function of the American Ceramic Society is primarily to promote the arts and sciences related to the silicate industries, it would seem that the Society should be interested in fostering the continuation of those branches of the industry

which have been launched as a direct result of the war. During the past four years we have watched with pride the development and improvement of a number of ceramic products which were hitherto not manufactured in this country, or, if so, were considered of inferior quality to similar products which could be imported from abroad. We need only mention in passing; the wonderful development of our optical glass industry; the perfection and production of the highest quality of chemical glassware; the establishment of a chemical porcelain industry; the manufacture of porcelain protection tubes for pyrometers; the remarkable improvement in the quality of American spark-plug insulators; etc.

The development and production of the above wares in this country was only brought about through the painstaking researches of our investigators in our laboratories and through the expenditure of much time and money by our manufacturers in surmounting the obstacles encountered in undertaking the manufacture of new products. It is only fair that these be rewarded for their efforts. Our manufacturers do not ask that preference be given their products over products of superior quality. They are, however, entitled to consideration in competition with products of equal or inferior quality. There has been a tendency in the past on the part of the public to prefer to pay a premium for wares bearing a foreign trade mark—in spite of the fact that similar wares of equal or superior quality of domestic manufacture could be secured. This has been true to a large extent in the purchasing of ceramic wares for our industrial, college and university laboratories. This tendency has not been encouraging to our manufacturers either in the improvement of their wares or in the development of new lines.

The ceramic industry is just approaching the dawn of its development in this country. If given encouragement, our laboratories and manufacturers will over-shadow the achievements of the past four years by the progress and development which is to come. Boost for the home products!

ORIGINAL PAPERS AND DISCUSSIONS.

STRUCTURE OF GRAPHITE IN RELATIONSHIP TO CRUCIBLE MAKING.[1]

By Reinhardt Thiessen.

When the American graphite crucible manufacturers were confronted with the necessity of using American graphite, altogether or in part, as a substitute for Ceylon graphite, on which they had previously depended almost entirely, it was soon discovered that crucibles containing American graphites were very unsatisfactory, as compared with those made of Ceylon graphite.

The problem was then raised as to why crucibles made of, or containing, American graphite flakes should not be as efficient as those made of Ceylon graphite; and, if possible, to find out how to utilize American graphite flakes in crucible practice.

In this investigation it was considered desirable to make some microscopic examinations of the structures of graphitè crucibles, as well as the graphite constituents themselves, and a microscopic examination, as well as a study, of the physical properties of the bonding clays used. Thirty-one samples of different makes of crucibles were examined, of both foreign and American makes.

It was soon found that the form and the structure of the graphite constituents, and the internal structure of the crucible itself, were very important factors in the successful manufacture of graphite crucibles. It is the purpose of this paper to present some of the results obtained during this investigation.

A brief discussion of the shape or form of the graphite particles will be first in order. The American graphites examined were ob-

[1] By permission of the Director, U. S. Bureau of Mines.

tained from Alabama, Pennsylvania and Canada and consist of thin plates or flakes, as they are generally called. These graphites are all similar, in that they are highly sub-laminated and may easily be reduced to still thinner flakes. The Alabama flakes are, generally speaking, the smallest and thinnest, ranging in size from that barely visible to the naked eye up to a width of about $1^1/_2$ to 2 millimeters. The Pennsylvania flake is slightly larger and thicker, and the Canadian flake averages larger than the Pennsylvania. The Madagascar flake is almost twice as large as the American flake. The size, largely due to comminution, is, nevertheless, strongly dependent on the innate structure of the graphite in question. The Alabama and Pennsylvania flakes are similar in form, as is shown in Figs. 1 and 2. There is a decided tendency towards circular and oval forms, although many irregular flakes are present in these two graphites. The Canadian flake is more irregular, although there is some tendency towards the circular and oval forms, as shown in Fig. 3.

The Madagascar flake, shown in Fig. 4, differs considerably in shape from the American graphite. Instead of a circular or oval outline, the flakes tend to be straight-edged, approaching the form of parallelograms or polygons. Many irregular forms are also present, as shown in Fig. 4. The Ceylon graphite particles are of an entirely different form, occurring in the shape of irregularly formed grains, with relatively few flake-like particles. This is shown in Fig. 5. Some of the particles are spherical and some are ovoid, while others are roughly lenticular, tetrahedral, cubical, or polyhedral with some sharp and many rounded corners and edges. Generally speaking, it may be said that the Ceylon graphite consists of irregular grains with rounded corners and edges. Cross sections of the graphite flakes and grains at the same magnifications are shown in Figs. 18 to 30.

The grain size of the Ceylon graphite, as used in making crucibles, is variable, ranging from those invisible to the naked eye to particles several millimeters in diameter or length. These sizes are largely a matter of grinding, screening and blending of the various grades thus produced, although the innate structure of the graphite particle itself exercises considerable influence.

There are, therefore, fundamental differences in the shapes and

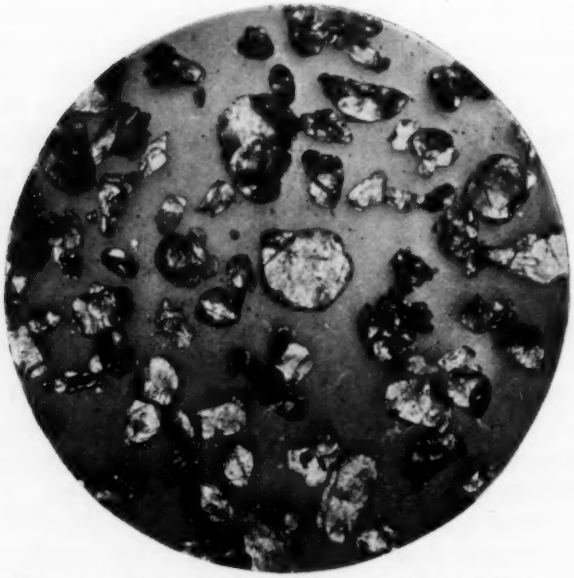

FIG. 1.—Alabama graphite flakes, seen flatwise, × 13.3.

FIG. 2.—Pennsylvania graphite flakes, seen flatwise, × 13.3.

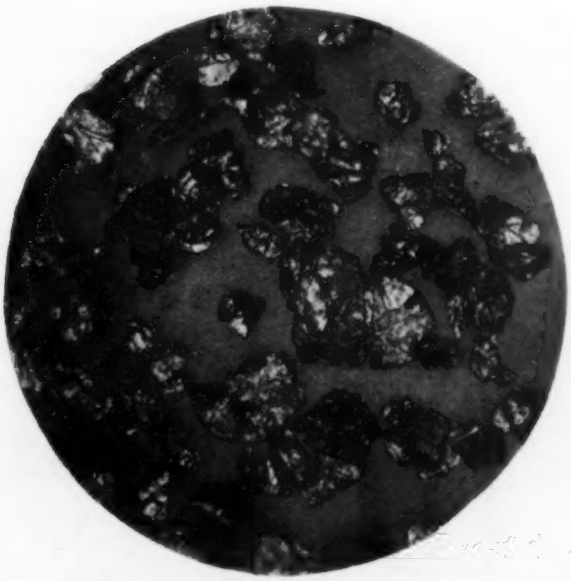

FIG. 3.—Canadian graphite flakes, seen flatwise, × 13.3.

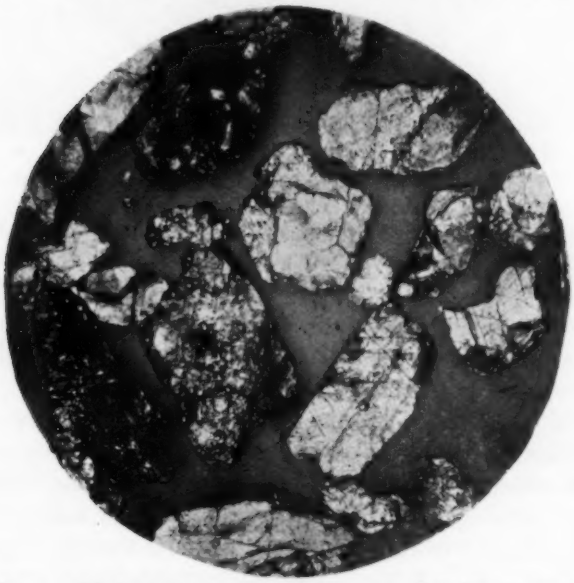

FIG. 4.—Madagascar graphite flakes, seen flatwise, × 13.3.

sizes of the American graphite flakes on the one hand, and of Ceylon graphite grains on the other hand, that naturally tend to cause great differences in their behavior under conditions of use.

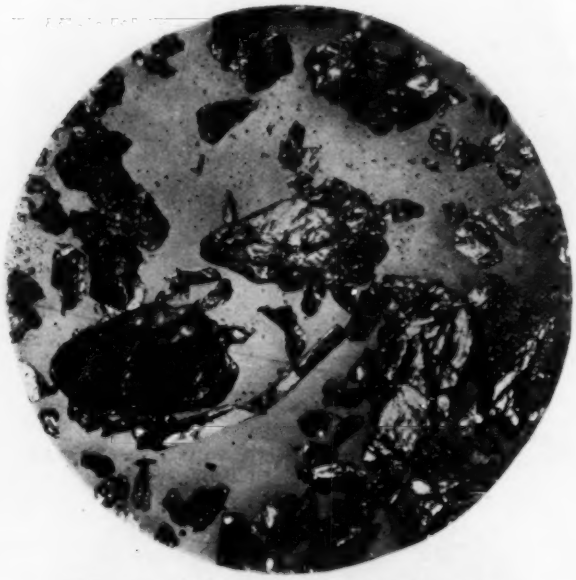

FIG. 5.—Ceylon graphite particles, × 13.3.

The Inner Structures of Graphites.

When seen in cross-section at a low magnification, the graphite constituents have a homogeneous appearance, with marks of laminations or other structures here and there. At higher magnifications, say at 200 diameters, there become visible various laminations, striations and divisions, blocking the graphite out into many forms and patterns.

All the American graphites under discussion show parallel laminations and sub-laminations into very thin sheets or flakes, the laminations being continuous along the whole length and breadth of the flake. With these graphites, the laminations are most pronounced in the Canadian flake and least pronounced in the Alabama flake. The Madagascar flake shows similar and

more definite laminations than the Canadian. See Fig. 6 (Canadian), Fig. 7 (Alabama), and Fig. 8 (Madagascar), all at 133 diameters. It will be noticed that the Alabama flake has rather rough surfaces, as seen in cross-section, compared with either the Pennsylvania, Canadian or Madagascar flake, giving, in general, a more irregular cross-section.

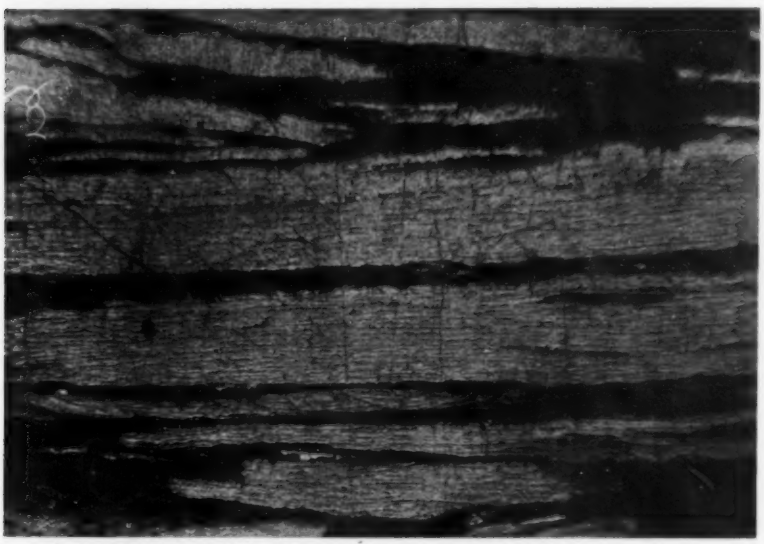

FIG. 6.—Cross section of Canadian graphite flakes embedded in clay, at a magnification of 133 diameters.

The Ceylon graphite has an entirely different structure, which may be subdivided into two main types. In the one (the least prevalent type) there is a parallel arrangement of very thin laminae. These are, however, not continuous from side to side of the grain, but are blocked off into more or less regular areas, when seen in cross-section, by means of breaks at right angles to the laminae (see Fig. 9, 133 diameters). The laminations are not necessarily parallel to the outer surfaces, as in the American flakes; in fact, they usually run diagonally from one surface to the other. Hence, the mass or grain is more or less regularly blocked off into forms approaching the parallelopiped and cube.

FIG. 7.—Cross section of Alabama graphite flakes embedded in clay, at a magnification of 133 diameters.

FIG. 8.—Cross section of Madagascar graphite flakes embedded in clay, at a magnification of 133 diameters.

FIG. 9.—Cross section of Ceylon graphite grains embedded in clay, at a magnification of 133 diameters.

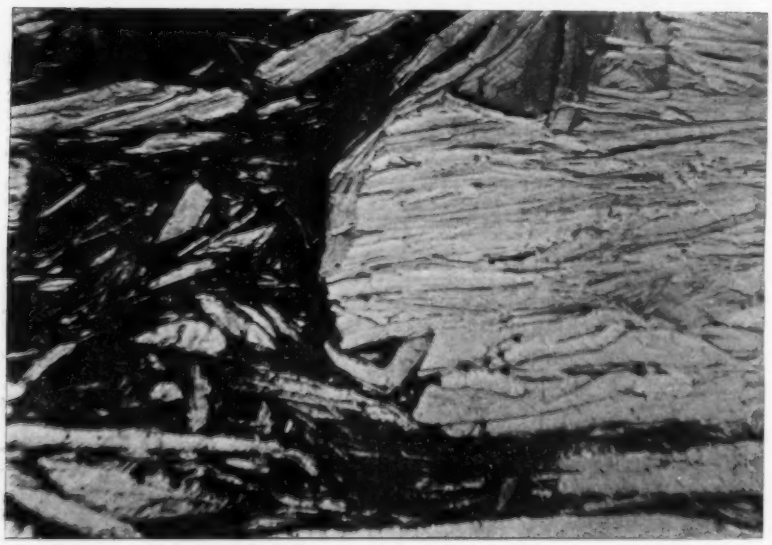

FIG. 10.—Cross section of Ceylon graphite grains embedded in clay, at a magnification of 133 diameters.

These sub-divisions are further composed of very thin plates, which are discussed later. In the other type of Ceylon graphite, which occurs more frequently, the grains are sub-divided into more or less regular triangular prisms and parallelopipeds. Figs. 10, 11 and 12 (133 diameters) represent this type. These prisms or blocks are further composed of very thin plates, parallel to each other, but variously arranged with respect to those of other prisms or blocks, as shown in the illustrations. A large variety of patterns are thus formed, due both to the different positions of the group of thin plates forming the prisms or blocks and the shapes of the prisms or blocks themselves.

This structure is vividly illustrated by the structure of the so-called graphitic acid. When Ceylon graphite is subjected to Brodie's reaction (oxidation with potassium chlorate and nitric acid), a yellow substance, called graphitic acid or graphitic oxide, is formed. The structure of this product is interesting. Even at the low magnification of 66 diameters (Fig. 13), it is shown to consist of groups of numerous thin plates closely packed and of a relatively few amorphous-appearing granules. When these are submersed in a dilute solution of ammonia, the whole mass apparently swells up, due to a slight separation of the thin plates from one another (Fig. 14). The mass thus loosened up is in a favorable condition for observation.

It is now clearly shown that the graphitic oxide "crystals" consist of the blocks or prisms, or parts of the same, that originally formed the graphite grains. The ultimate plates which formed such aggregates have been impregnated, partially separated, and have become translucent, or transparent, through the reaction. Under the ultra-microscope these plates are shown to be composed of ultra-microscopic particles or micellae closely cemented together in the same manner as clay, except that the clay particles are very much larger. They must, therefore, be considered colloidal rather than crystalline.

The grains or particles of the Ceylon graphite are, therefore, very complex objects. No matter into how small sub-divisions the grains are broken, nor in what direction they break, the fracture is apt to be an irregular grain and rarely a flake.

FIG. 11.—Cross section of Ceylon graphite grains embedded in clay, magnified 133 diameters.

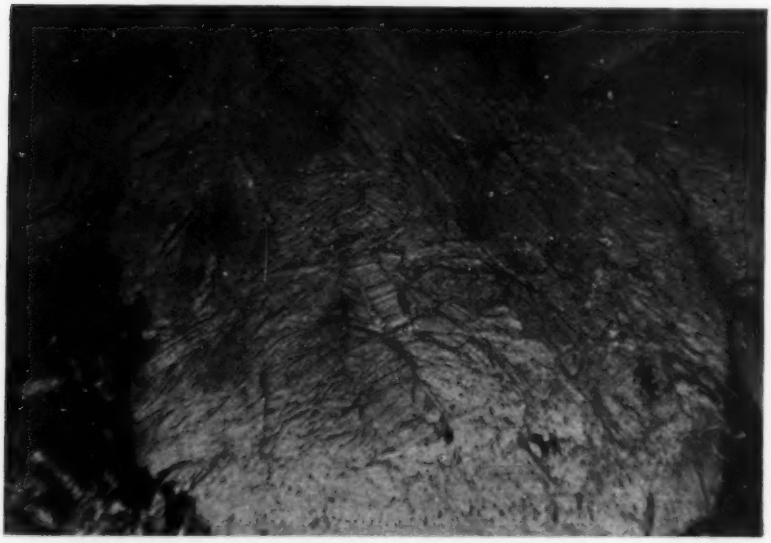

FIG. 12.—Cross section of Ceylon graphite grains embedded in clay, magnified 133 diameters.

FIG. 13.—"Graphite oxide" floated out in ammoniacal water. Many groups of thin plates are distinguishable, × 66.

Structures Observed at High Magnifications.

When sections of graphite are cut at right angles to the laminae and observed at a magnification of from 1000 to 2500 diameters, the laminae shown at a magnification of 133 diameters (Figs. 9 to 12) are found to be still further sub-divided into sub-laminae. Fig. 15 (666 diameters) shows this in a Canadian graphite flake. It will be noticed (Fig. 15) that the photograph shows a number of bands quite definitely marked off, and that each of these bands is further sub-divided into a considerable number of less definitely marked bands. Each of the faintly marked bands represents a thin plate. When graphitic acid is formed from graphite, a more or less complete separation of the plates is effected. Since the magnification of Fig. 15 is 666 diameters, direct measurement will readily show that the more definitely marked plates are from 5 to 10 microns thick, while the less definitely marked plates are in the neighborhood of only 1 micron in thickness

All the graphites examined reveal a similar layering under high magnification, provided the sections are cut at right angles to the laminae. In view of the very many positions, a compilation of such plates may assume in the Ceylon graphite, sections of the prisms in every position from cross-sections to parallel sections are met with in this variety, as shown in Figs. 10 and 12.

FIG. 14.—Two groups of graphitic oxide plates at a higher magnification. In the one the plates are seen edgewise and in the other flatwise, × 66.

Very often structures are revealed that have somewhat the appearance of crystalline particles or granules. The definiteness of the graphite structure, the shape and size of the plates, the compactness of the mass, the close or loose packing of the particles, all vary considerably in the different graphites examined, as well as in the different flakes or grains of the same graphite, although each kind possesses characteristic qualities.

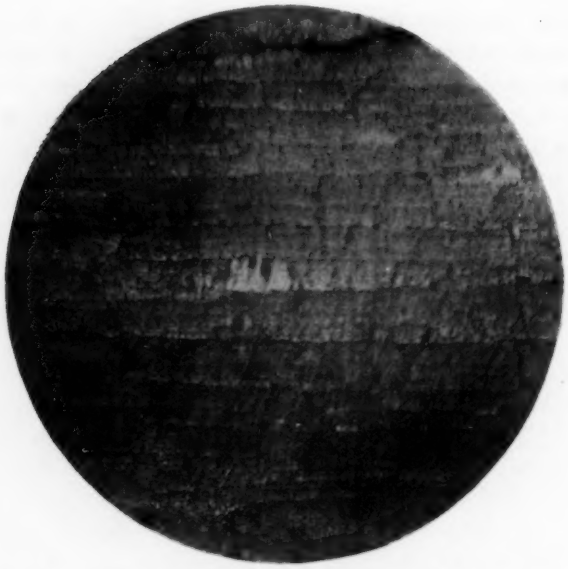

FIG. 15.—Cross section of a Canadian graphite flake at a high magnification, × 666.

FIG. 16.—Cross section of an Alabama graphite flake at a high magnification, × 666.

Fig. 16 (x 666) shows a section of the Alabama flake and represents an appearance frequently found. It will be seen that the mass, which appears amorphous at a magnification of 200 diameters, is here resolved into very minute particles of a crystalline appearance. Fig. 17 (x 666) represents a section of the Madagascar flake. It is noticeable that the crystalloid particles are here considerably smaller than in the Alabama flake. In fact, in Fig. 17, they are so small that they are just within the limit of visibility. In other cross-sections, however, these particles approach those of the Alabama flakes in size. The arrangement, size, form and proximity to one another of the particles in the Ceylon graphite vary considerably, but, in a general way, are very similar to those of the other graphites.

The question now arises whether or not these particles are really crystalline. Many of them appear completely homogeneous at any magnification that they were subjected to. Very many, on the other hand, are resolved into ultra-particles or micellæ, and appear non-crystalline. It is very probable that all the particles resolve into ultra-particles under proper illumination and lenses of high resolving power. This supposition is borne out by the ultra-structure of graphitic acid.

The Arrangement of the Flakes in the Crucible.

An English crucible, in which all the graphite is Madagascar flake, has been pointed out to be one of the most durable pots on the market. The structure of the body of this make of crucible will be taken up first and will serve as a standard of comparison. A cross section of this crucible is shown in Fig. 18 at a magnification of 13.3 diameters. The white linear patches represent the cross sections of the graphite flakes; the darker rounded or irregular areas represent the sand particles present in most graphite crucibles as one of the ingredients, while the grayish mass between these two constituents represents the clay binder and the clay mixed with finer particles of graphite.

It will be noticed that the flakes are arranged, in general, parallel to each other, and approximately parallel to a line tangent to the outer surface of the crucible-wall, in quite uniform and regular order. In a measure, the flakes overlap each other, shingle

FIG. 17.—Cross section of a Madagascar graphite flake at a high magnification, × 666.

FIG. 18.—Cross section of a portion of a crucible wall in which the graphite is all Madagascar flake, × 13.3.

fashion. Only occasionally is this order departed from. Occasionally, also, some of the flakes are grouped more or less, but this grouping is slight. The flakes themselves are of a rather uniform shape and of uniform sizes, grading gradually from the largest to the smallest.

It is noticeable, also, that the flakes are closely packed together. This feature is well shown in Fig. 8, representing cross sections, at a magnification of 133 diameters, of the medium to the smaller flakes. The uniformly parallel arrangement in general, and the regular plate-like shape of the flakes are the prominent features in the structure of this crucible.

The sand in this crucible is shown to be irregularly distributed. Fig. 18 reveals but a few particles of sand, which are rather variable in size and sharp cornered.

A sample of an American make of crucible, containing Canadian flakes only, is shown to be similar in structure and in the arrangement of the flakes to the English make. The uniformity in arrangement and the parallel layering of the graphite flakes appears to be even better than that in the English. The shingle-like overlapping is quite apparent in Fig. 19.

While the flakes of the Canadian graphite are similar to the Madagascar flakes in form, it will be noticed that a certain number of them are slightly lenticular. The flakes in this sample are, however, not nearly as closely packed as those in the English make of crucible. This is clearly shown in a comparison of Figs. 18 and 19. This observation is in harmony with the fact that the percentage of graphite, *viz.*, 40 per cent, is rather low. The sand particles in this sample are comparatively small, but well distributed through the body.

The structure of a Japanese crucible, the source of the graphite flakes of which has not been ascertained, is also similar to that of the English make of crucible. Although the general shape of the flakes is similar to those of the two foregoing samples, they are not quite of the same uniformity of shape; also, the range of sizes is greater, a considerable number of the flakes being both broader and thicker, but the plate-like shape is retained in general. The sand particles are large and rounded and well distributed through the body. The flakes appear to be quite closely packed, closer than

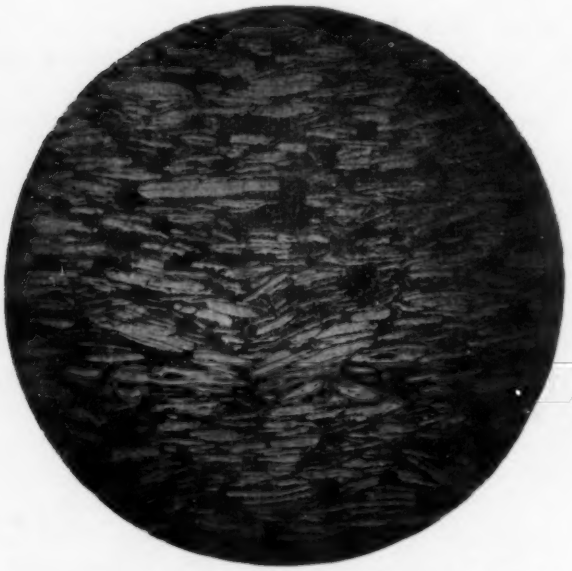

FIG. 19.—Cross section of a portion of a crucible wall in which the graphite is all Canadian flake, × 13.3.

FIG. 20.—Cross section of a portion of a Japanese crucible wall, graphite unknown, × 13.3.

the sample containing Canadian graphite, but not as close as in the English sample (see Fig. 18).

The cross section of the wall of a crucible, containing Ceylon graphite only, presents an entirely different appearance. In this, there is not nearly that systematic and uniform arrangement of the graphite constituents as has heretofore been the rule. In fact, great irregularity is noticed. In some crucibles the flakes are jumbled together in a chaotic condition, while in others there is a fairly good systematic arrangement. These conditions are fairly well represented in Figs. 21, 22 and 23, and represent about all the varieties of conditions of all the pots containing all Ceylon graphite. Fig. 21 shows hardly any orientation of the graphite constituents; the angularity, irregularity, the isodiametric qualities, and the great range in sizes of the particles, stand out prominently, and are at once noticeable. In Fig. 22, there appears a considerable degree of orientation. Notice that the particles are, on the whole, more plate-like than those shown in Fig. 21. The great range in sizes is also particularly noticeable here, a very large proportion of the graphite being in the form of fine dust. The condition shown in Fig. 23 differs in that it contains a relatively large proportion of large particles and a relatively small amount of the finer matter.

Fig. 24 represents a section of a crucible in which there is 55 per cent graphite, 25 per cent of which is said to be Canadian flake. The Canadian flake may readily be distinguished from the Ceylon through the difference in shape.

Figs. 25 and 26 represent sections of crucibles in which 90 per cent of the graphite is Ceylon and 10 per cent is Pennsylvania. The Pennsylvania flake here again is easily distinguishable from the Ceylon graphite. The bodies of these crucibles were of poor consistency and brittle, giving rise to unfinished surfaces, as may be seen in the figures.

Fig. 27 represents a section of a crucible reported to contain 51 per cent graphite, 75 per cent of which was said to be Ceylon and 25 per cent to be domestic flake.

Fig. 28 shows a similar appearance; yet the nature and arrangement of the flakes, which are evidently Alabama, differ.

Fig. 29 represents a cross section of a crucible containing 52.5

FIG. 21.—Cross section of a crucible wall in which the graphite is all Ceylon, × 13.3.

FIG. 22.—Cross section of a portion of a crucible wall in which the graphite is all Ceylon, × 13.3.

FIG. 23.—Cross section of a portion of a crucible wall in which the graphite is all Ceylon, × 13.3.

FIG. 24.—Cross section of a portion of a crucible wall containing a total of 55% graphite, 75% of which is Ceylon and 25% Canadian flake, × 13.3.

FIG. 25.—Cross section of a portion of a crucible wall in which 90% of the total graphite is Ceylon and 10% Pennsylvania flake, × 13.3.

FIG. 26.—Cross section of a portion of a crucible wall in which 90% of the total graphite is Ceylon and 10% Pennsylvania flake, × 13.3.

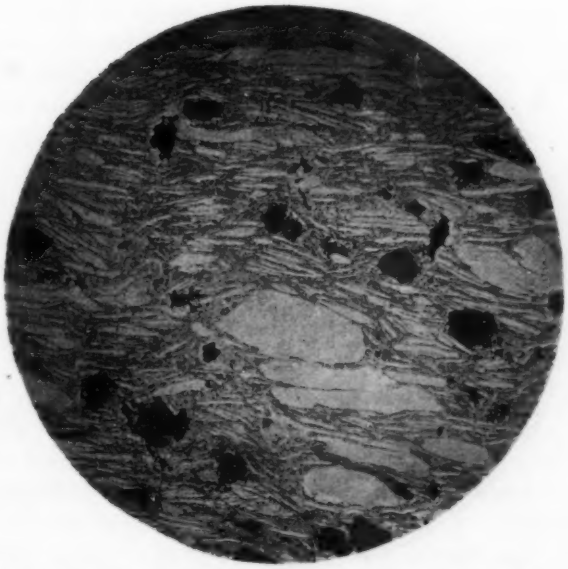

FIG. 27.—Cross section of a portion of a crucible wall said to contain a total of 51% graphite, 75% of which is Ceylon and 25% domestic flake, × 13.3

FIG. 28.—Cross section of a portion of a crucible wall, in which 83.5% of the total graphite is reported to be Ceylon and 17.5% domestic flake, × 13.3.

FIG. 29.—Cross section of a portion of a crucible wall containing 52½% graphite, 85% of which is Ceylon and 15% Madagascar flake, × 13.3.

FIG. 30.—Cross section of a portion of a crucible wall in which 65% of the total graphite is Ceylon, 20% Alabama flake and 15% Madagascar flake, × 13.3.

per cent graphite, 85 per cent of which is Ceylon and 15 per cent of which is Madagascar flake. The large differences in the sizes of the Madagascar flakes shown in the figure, over that of the Alabama and the Pennsylvania flakes, shown in the previous figures, is clearly evident.

Fig. 30 represents a section of a crucible in which the graphite consists of 65 per cent Ceylon, 20 per cent Alabama flake, and 15 per cent Madagascar. Here also the three kinds of graphite constituents are distinguishable.

The Effect of Graphite Characteristics on the Graphite Crucible.

Having analyzed the relative sizes, forms and structures of the graphite constituents and the structure of the crucible itself, it now remains to correlate the findings with the essential question of the crucible problem, as stated at the outset. Unfortunately, not enough data are at hand to give a definite answer. This part of the investigation, as in all other phases relative to this problem, should be looked upon as an entering wedge towards the solution of a most important commercial problem. It is hoped that all crucible manufacturers will use these data and study their ware in a similar way, and connect and correlate the data with an aim towards the improvement of their product and finally make the data and findings available for the trade in general.

A few suggestions and conclusions of the findings, nevertheless, may be briefly outlined.

In looking over the cross sections of the walls of different crucibles, various factors that entered into the structure of these became apparent. The factors follow:

(*a*) The arrangement of the flakes.

(*b*) The form of the flakes.

(*c*) The different sizes of the flakes; in other words, whether fine or coarse, as a whole.

(*d*) The large range of sizes.

(*e*) The proximity of the graphite constituents.

(*f*) The total surfaces of the constituents in relationship to their volumes.[1]

(*g*) The physical conditions of the clay binder and the sand and other ingredients that are invariably found in the make-up of the crucible.

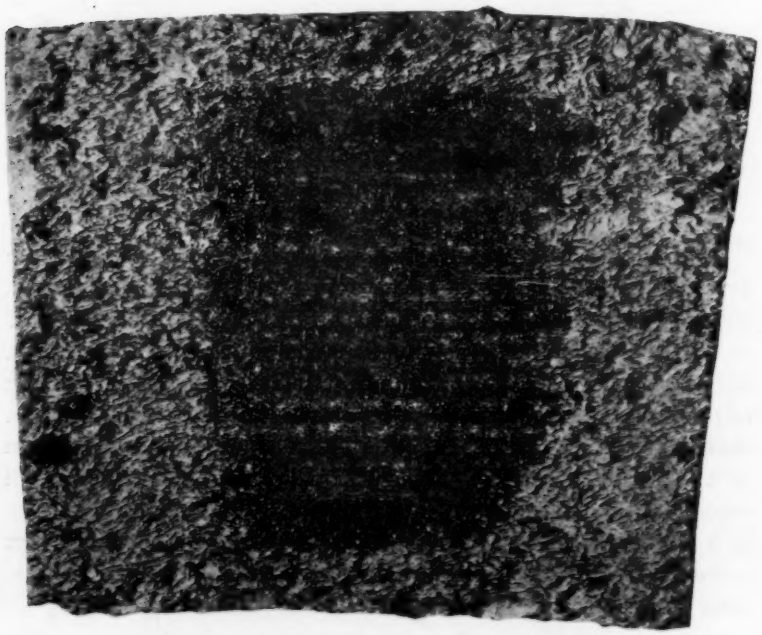

FIG. 31.—Cross section of a block sawed from the wall of an English crucible containing Madagascar graphite flake only, a cross section of which is shown in Fig. 18, subjected to a temperature of approximately 1250° C. in a muffle furnace, for $7^1/_2$ hours. (× 3.)

An important factor in the efficiency of a crucible appears to be the arrangement of its graphite. Theoretically, everything else being equal, a pot in which the graphite is in the form of flakes arranged parallel to each other and parallel to a tan-

[1] Bleininger, A. V., "Recent Developments in Ceramics," *J. Ind. Eng. Chem.*, **10**, 844–849 (1918). Bleininger, A. V., "Notes on the Crucible Situation," *J. Am. Inst. Metals*, **11**.

gent to the outer wall of the pot, should be the most resistant to oxidation under the conditions to which the crucible is subjected during use. A few experiments will readily verify this theoretical conclusion. Cubical blocks were sawed out of various makes of crucibles and all were subjected, as far as possible, to the same conditions in a muffle furnace heated to a temperature of approxiamtely 1250° C for $7^1/_2$ hours. In every case, excluding other factors, it was found that oxidation was least on the sides parallel to the flakes or flat-sides of the graphite grains and most on the sides perpendicular to the flakes or longer axes of the graphite constituents. The English make of crucible, containing all Madagascar flakes (a cross section of which is shown in Fig. 18), shows these results most emphatically. Fig. 31 shows the interior of a block cut out of the same crucible and exposed to the aforesaid conditions. The outer, light zone represents the burned-out part, while the dark inner area represents the unburned portion. It will be noticed that the graphite has burned out the least on the sides of the block that formed the inner and the outer surface of the crucible wall; that is, on the sides parallel to the flakes, while on the sides that were sawed out of the wall, the sides perpendicular to the flakes, the graphite has burned out to a depth almost three times as great.

The same phenomenon, although not nearly as pronounced, is shown in the crucible containing all Canadian flake, a cross section of which is shown in Fig. 19. Fig. 32 shows a cross section of a block of this crucible subjected to exactly the same conditions as the other. Here, also, the graphite has been burned out the least on the sides that formed the outer and the inner surfaces of the wall of the pot, or the sides parallel to the largest surfaces of the flakes, while it is burned out to the greatest depth on the sides perpendicular to the flakes. It will be noticed, however, that in this case the graphite had not been burned out nearly as deep as in the previous case (the English crucible). Another clear-cut example is shown in a crucible containing 83.5 per cent Ceylon graphite and 17.5 per cent Alabama flake, shown in cross section in Fig. 28. Fig. 33 represents the interior section of a block of this crucible subjected to the same conditions as the others; and again, the sides parallel to the flakes have oxid-

ized the least and the ones perpendicular to the longer axes, the most, but very much deeper, on either side, than any of the specimens heretofore shown.

If now a hypothetical case is taken in which all the graphite

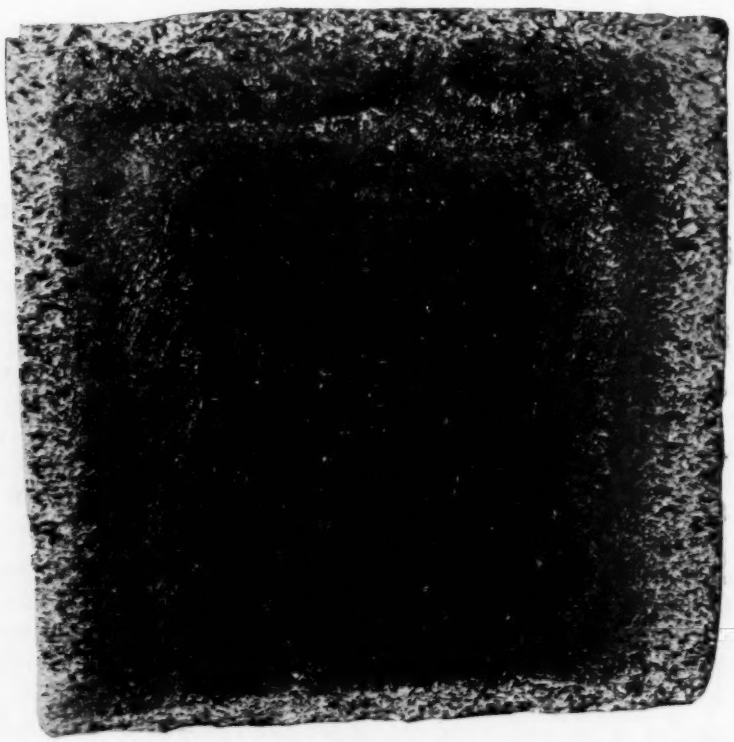

FIG. 32.—Cross section of a block sawed out of a crucible wall containing Canadian flake only, shown in cross section in Fig. 19, and subjected to a temperature of approximately 1250° C. for $7^1/_2$ hours. (× 3.)

flakes, any Canadian flakes, were arranged perpendicular to the tangent to the outer wall of the crucible, all other factors being disregarded, the whole wall of the pot would be oxidized through and through under a few heats under similar conditions to which these sample blocks were subjected; or, if the crucible contained

only Ceylon graphite consisting of relatively large grains of approximately uniform diameter, the same thing would happen.

FIG. 33.—Cross section of a block sawed out of a graphite crucible wall, shown in cross section in Fig. 28, and subjected to a temperature of approximately 1250° C. for $7^1/_2$ hours. (× 3.)

Other Factors Involved.

The great differences in the depths to which oxidation of the graphite has penetrated leads to the conclusion that other factors are involved. One such factor is easily discovered in the fineness or coarseness of the constituents, a factor which connects up directly with the foregoing. This is shown in a comparison of the examples already given. The specimen containing the Canadian flake (Figs. 19 and 32), in which the texture is relatively fine, should be compared with the English specimen containing all Madagascar flake (Figs. 18 and 31) and the one shown in Figs. 28 and 33, in which the textures are considerably coarser. The relatively greater depth of oxidation is clearly seen.

To go one step further in this direction, Figs. 34 and 35 are

offered for examination, both from pots in which the graphite consists of 90 per cent Ceylon and 10 per cent Pennsylvania flake and shown in cross sections in Figs. 25 and 26. This material is still coarser than any previously shown. In Fig. 34, the graphite

FIG. 34.—Cross section of a block sawed out of a crucible wall containing 90% Ceylon and 10% Pennsylvania flake, also shown in cross section in Fig. 25, and subjected to a temperature of approximately 1250° C in a muffle furnace for $7^1/_2$ hours. (× 3.)

is seen to be almost completely burned out, only a small core being left in the center. In the one shown in Fig. 35, the coarsest

of them all, the graphite is burned out completely. In Fig. 34, the rule of burning out most on the side perpendicular to the longer axis is followed quite closely.

FIG. 35.—Cross section of a block sawed out of a crucible wall in which the total graphite consists of 90% Ceylon and 10% Pennsylvania flake, also shown in cross section in Fig. 25, and subjected to a temperature of approximately 1250° C in a muffle furnace for 7½ hours. (× 3.)

It is safe to say that the sizes of the graphite constituents, especially the graduation of sizes (that is, whether the particles are, on the whole, coarse, medium or fine and whether there is a uniform graduation of sizes from the smallest to the largest) play an important part in the resistance to oxidation; but there are other

factors involved that must first be eliminated and their influence determined, each for itself, before any definite statements can be made.

The Plasticity of the Clay Used as a Bond.

The plasticity of the clay is of the utmost importance, and upon this the plasticity of the graphite and clay mixture must largely depend. But the clay by no means determines this alone, since the plasticity depends also upon the form of the graphite constituents used.

In a mixture of clay and graphite particles, in which the latter are in the form of grains of a more or less uniform diameter, and in which the sizes graduate more or less uniformly, the plasticity of the clay, while it is somewhat reduced, is reduced to a minimum degree. The mixture, under pressure, will flow in any direction without rupturing. In a mixture in which the graphite is in the form of flakes or thin plates, the plasticity will naturally be reduced to a far greater degree, because the mass is not equally free to flow in all directions under pressure, being interfered with to a larger degree in the direction perpendicular to the plane of the flakes than in any other, and being least checked in the direction parallel to the flakes. The flow will naturally tend to go in this latter direction. Any movement in the direction perpendicular to the flow of the flakes will result in a disarrangement of any parallel layering, and tend to shear and rupture the bond, while a movement in the direction parallel to the flakes will tend to cause too great slipping while the pot is being molded. A mixture with Ceylon graphite (grains instead of flakes) will, for these reasons, be more uniformly plastic, more easily brought into the proper form and with less danger of rupturing than a mixture containing graphite flakes.

Peptization of Clays.

Clays as used in crucible making must first be very finely ground. But no matter how finely ground they may be they are mostly in the form of particles. Under the microscope most of these appear as coarse particles. Clays in this form, after being mixed and kneaded with water, are still largely in the form of

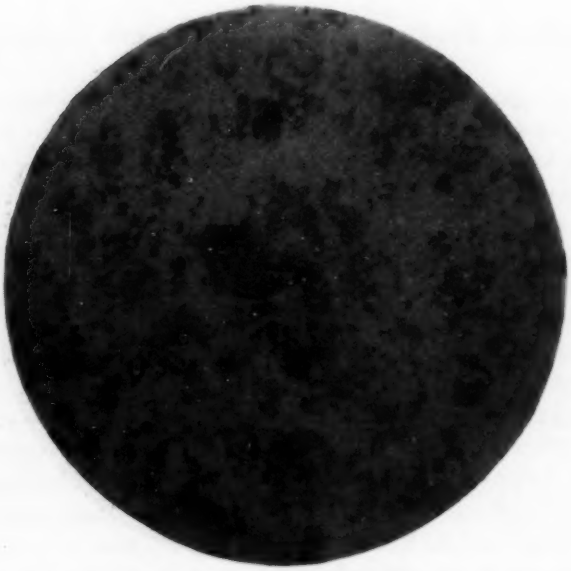

FIG. 36.—Unpeptized clay, graphite and water mixture, from which the graphite has largely been removed. (× 166)

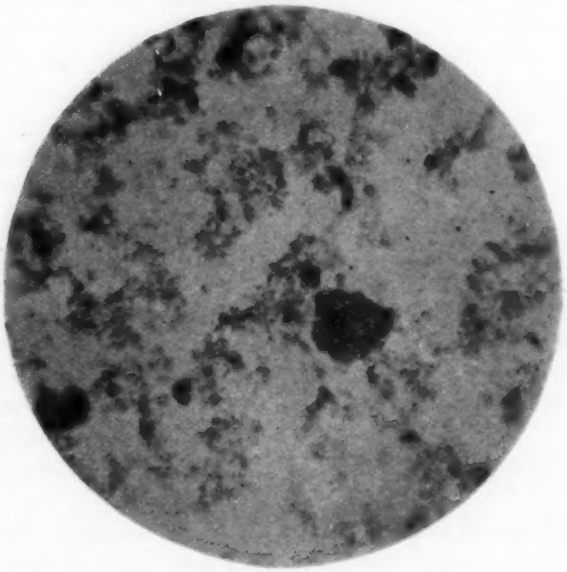

FIG. 37.—Unpeptized clay, mixed and kneaded with water. (× 133.)

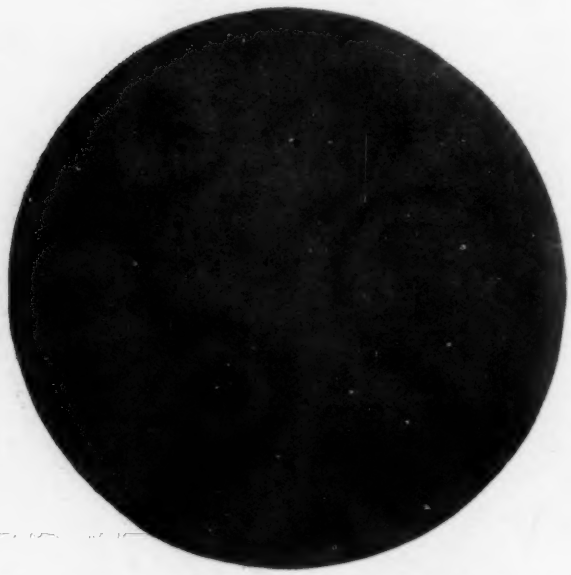

FIG. 38.—Peptized clay, graphite and water mixture from which the graphite has been largely removed. (× 133.)

FIG. 39.—The ultra clay particles from the mixture, shown in Fig. 38. (× 1666.)

coarse particles and are only partially reduced to their ultra-particles.

It is possible, however, to disperse clays completely by means of proper reagents; in other words, they may be separated into their ultra-particles, or peptized, as shown in Figs. 38 and 39.

The specific surface of clay, when thoroughly dispersed, has been increased enormously over that of the unpeptized clay or clay in only a partially dispersed condition. The specific surface in the graphites is an important factor in graphite-crucible efficiency. The more plate-like and the thinner the flakes the greater is the specific surface. Hence, the total surface of the Alabama flake graphite over that of the Ceylon graphite must be very large. However, this greater surface can be more than counter-balanced by thoroughly dispersing the clay. A closer proximity and a more tenaceous adherence of the graphite flakes to one another is then made possible and, at the same time, the flakes are free to move or slip over one another due to the lubricating action of the film of finely-divided clay particles covering each graphite flake.

Fig. 37 shows the original clay (Dorset) mixed and kneaded with water. Fig. 36 shows an unpeptized clay and graphite mixture, from which the graphite has largely been removed. Fig. 38 shows a mixture of peptized clay and graphite, the graphite having largely been removed. Fig. 39 shows the ultra-particles of the clay at a very high magnification.

Summary.

Many crucibles containing Ceylon graphite were examined; also, an English crucible containing Madagascar graphite, several American crucibles containing American graphite, and one Japanese crucible (graphite origin unknown) were examined.

The American graphites (Canadian, Alabama, and Pennsylvania) and the Madagascar graphite have a flake formation, while Ceylon graphite has a more granular form.

Some factors entering into the structure of graphite crucibles follow:

(*a*) If the graphite is in the flake form, the flakes should be parallel to each other and parallel to the tangent to the outer

wall of the crucible. This arrangement tends to minimize oxidation. More care must be used in making crucibles containing flake graphite, as the clay-graphite mixture will not flow in all directions, due to the unsymmetrical shape of the admixed graphite particles.

(*b*) It is better to have the graphite particles uniformly small, rather than uniformly large. If mixed sizes are present, it is advantageous to have an even graduation from the largest to the smallest.

(*c*) The graphite particles should be uniformly and closely packed together.

(*d*) The total surface of all constituents, in relationship to their volume, is a factor that has long been recognized.

(*e*) The clay should be in a suitable physical condition to make it plastic even after the addition of the graphite.

Acknowledgments.

The writer is deeply indebted to Mr. F. L. Arnsberg and Mr. Arthur J. Jackman of the Vesuvius Crucible Company, for their willing and enthusiastic coöperation, and for imparting their valuable experience, thus being of great help in the work. The Vesuvius Crucible Company and Mr. George D. Dub also rendered valuable assistance in furnishing much of the material required.

The writer wishes further to express his gratitude to Mr. D. A. Lyon, Supervisor of Stations, and Mr. A. C. Fieldner, Supervising Chemist, who did much to facilitate the work; and also to Mr. R. T. Stull, Superintendent of the Ceramic Station of the Bureau of Mines at Columbus, Ohio.

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PITTSBURGH, PA.,
May 9, 1919.

THE COOLING OF OPTICAL GLASS MELTS.

By Howard S. Roberts.

When a melt of glass is removed from the melting furnace, it may be either left to cool in the pot, or be cast and rolled into a plate. This paper has to do with the former case, where the melt is cooled in the pot.

The choice between these two methods is a question of the quality of glass desired and of expediency. When the melt is set out of the furnace it is never entirely homogeneous. The middle is usually free from contamination, but near the bottom and sides of the pot, the glass is badly contaminated with material dissolved from the pot. If the melt is cooled in the pot, this contaminated glass need not be much disturbed, but where it is cast and rolled into a sheet, it is apt to be spread rather evenly through the whole plate.

While the process of cooling in the pot involves a greater amount of labor and waste in the mechanical preparation of the glass, it can be made to yield glass of the highest quality. The rolling process, on the other hand, may give a large yield of glass that is entirely suitable for many, if not most, optical instruments.

Where the melt is cooled in the pot, the conditions to be sought are: (1) that the cooled melt shall break into large, reasonably rectangular blocks, having smooth faces; and (2) that not much glass shall be spoiled by inhomogeneity, *i. e.*, by striae, bubbles, crystals or devitrification.

It is not feasible to cool the melt so slowly that it will remain unbroken; nor is this particularly desirable because of the necessity of breaking it up later for examination and for rejection of defective portions.

At the melting temperature, glass is a fairly mobile liquid. As it cools its viscosity increases continuously, until at ordinary temperatures, though probably not infinite, the viscosity is so great that we apparently have an elastic solid. For convenience

we shall divide the whole range of cooling into three stages: The Stage of Fluidity; The "Annealing Range;" The Final Stage.

Stage of Fluidity.

During this stage, the glass is apt to be spoiled by striae due to convection; by the formation of vacuum bubbles, and, in certain glasses, by the formation of one or more crystalline phases in the glass itself.

Convection.—On leaving the furnace, the glass is still quite fluid, having been stirred until the time of setting out. The portion close to the sides and bottom of the pot is richer in material dissolved from the pot and has an index of refraction different from that of the glass nearer the center. As the melt cools convection currents tend to draw streamers of this contaminated glass upward through the middle of the melt. Below the "setting-out temperature" the glass is too viscous to readily assimilate these streamers by solution; so that, having once appeared, they remain as "striae" or "ream." Particles of foreign material, such as bits of the pot, may also be carried to the middle of the melt where they usually float upward or settle, dragging tails of contaminated glass behind them. Convection continues at a decreasing rate as viscosity increases on cooling, and soon becomes inconsiderable.

As convection is caused by the settling of denser, cooler glass, it can be reduced by insulating the upper portion of the melt, and cooling rapidly from the bottom. Unfortunately, present melting practice usually insures that the bottom of the pot, on leaving the furnace, shall be at a somewhat higher temperature than any other portion; moreover, the bottom is usually made much thicker than the sides. Thus it has not been found possible entirely to prevent convection even by immersing the bottom of the pot in a pan of water.

It should be emphasized that measures to reduce convection should be taken immediately on setting the pot out of the furnace. The surface of the melt should immediately be covered with a layer of kieselguhr or other light insulator; and the pot be placed on bricks as quickly and with as little agitation of the glass as possible.

Vacuum Bubbles.—Another phenomenon peculiar to this stage is the formation of vacuum bubbles. These are caused by the premature "setting" of the top surface of the melt, thus enclosing the inner portion in a rigid envelope. On further cooling, the contraction of the interior greatly enlarges any small bubbles that may be present and may even draw in considerable quantities of air through cracks in the pot. This effect can be entirely prevented by insulating the top surface of the glass sufficiently to prevent the temperature of that portion from falling much below the temperature of the center of the melt.

Crystallization.—While the glass is yet soft, conditions are particularly favorable for the formation of crystalline phases in the melt, giving rise to the phenomenon of "crystallization" or "devitrification."[1] In addition, undissolved particles of sand or of the pot may form nuclei for the deposition of crystalline material from the glass. Given time enough at a suitable temperature, one or more crystalline phases may separate from any glass, but this effect begins reluctantly and proceeds slowly, so that most glasses can be cooled rapidly enough to prevent it.

Conditions to be Secured during Stage of Fluidity.—To inhibit convection and the formation of bubbles, the surface of the glass should be kept warm and heat removed chiefly from the bottom. The cooling should be made rapid down to a temperature below that at which there is no further danger of crystallization or devitrification. Convection can be further reduced by intensive chilling of the bottom of the pot by means of a current of compressed air or a fine stream of water, during the last hour before it is removed from the melting furnace.

Annealing Range.

The rate of cooling through the second stage determines the size and shape of the pieces into which the melt will break. The whole of the cooled melt, except the portion close to the pot, should be found broken into large, reasonably rectangular blocks, having smooth, flat surfaces through which their interior may be examined. Under these conditions the blocks have been

[1] N. L. Bowen, *J. Am. Ceram. Soc.*, 1, 599 (1918).

found to break clearly, but where the blocks have curved surfaces, they tend to form wedge-shaped fragments having very rough surfaces when broken. In extreme cases the interior portion of the melt may be found in the form of a large, flattened sphere of glass called a "marble" or "onion," which can seldom be broken satisfactorily.

When cold, the melt is usually found to be cut by cracks of two types: curved cracks, roughly spherical and concentric with the center of the melt, which form shells an inch or two thick in the outer portion of the melt; and plane cracks which divide the melt into large blocks.

Spherical Cracks.—If, as must always be the case, different parts of the melt cool at different rates, the resulting differences in rate of contraction cause stresses in the melt, which in turn tend to make the glass flow in such a way as to relieve the strains set up by them. The velocity of this release of strain decreases as the temperature falls, because of the increase of viscosity on cooling, and is, at any instant, nearly proportional to the total amount of strain causing it.[1] Consequently the amount of strain present will tend to approach such a value that it will be relieved, by internal flow, at the same rate as that at which fresh strain is introduced. As this flow is not very rapid, even at high temperatures, and is imperceptible at low temperatures, strain persists long after fresh strain ceases to be introduced.

Rather than consider the differences between cooling rates, it is somewhat simpler to speak of the time rate of change of the temperature drop (since the difference between the rates of change of two variables is equal to the rate of change of their difference, this amounts to the same thing). Then, if we assume that the coefficient of linear expansion of the glass is constant over the maximum temperature range throughout the melt at any instant, the differential rate of contraction, that is the time rate at which strain is introduced, is everywhere proportional to the time rate of change of the temperature gradient.

When the melt is set out of the melting furnace, there is little temperature difference through it. On account of convection

[1] This question is fully discussed in a paper in preparation by L. H. Adams and E. D. Williamson.

and, perhaps, the transfer of considerable quantities of heat through the glass by radiation as well as by conduction, the temperature drop between the center and the outside of the glass does not reach a maximum until the melt has cooled to a low red heat. An idea of this can be secured from Fig. 7, following.

After passing the maximum this temperature drop decreases, and disappears when the melt has cooled to constant temperature.

While the temperature drop is increasing the outer portions of the melt are contracting more rapidly than the inner and the strain consists of radial compression throughout, accompanied by tangential tension in the outer portions and tangential compression in the inner.[1]

After the maximum drop is passed, the inner portions are contracting more rapidly than the outer, and the stresses being introduced consist of radial tension throughout, accompanied by tangential compression in the outer portion and tangential tension in the inner. As the strains now being introduced are the reverse of those introduced before the temperature drop reached its maximum, their first effect is to neutralize the residuum of the strain introduced at that earlier time. On further cooling and reduction of the temperature drop strain builds up again and tends to fracture the glass by curved cracks more or less normal to the direction of the forces of radial tension.[2] During the earlier part of the annealing range, as has been said, strain is relieved by flow of the glass so much more rapidly than at lower temperatures that it does not accumulate sufficiently to cause cracks. Thus the spherical cracks are entirely due to the later decrease of temperature gradient.

This tendency to fracture increases as we recede from the center,[2] therefore fractures tend to follow one another, as strain increases, from the outside toward the center of the melt. If no other factor appears, the final form of the melt is a roughly

[1] We here assume that the isothermal surfaces in the melt are spheres. Actually they appear to be roughly spheroidal, approaching the spherical form at the center.

[2] The question of the elastic stresses produced by temperature differences in a solid sphere has been discussed by E. D. Williamson, *J. Wash. Acad. Sci.*, **9**, 209–217 (1919).

spherical core, or "marble," of glass surrounded by a series of shells very like those of an onion. The size of this core depends on the course of the temperature gradient.

This effect is shown in Fig. 1, which is a photograph of a melt

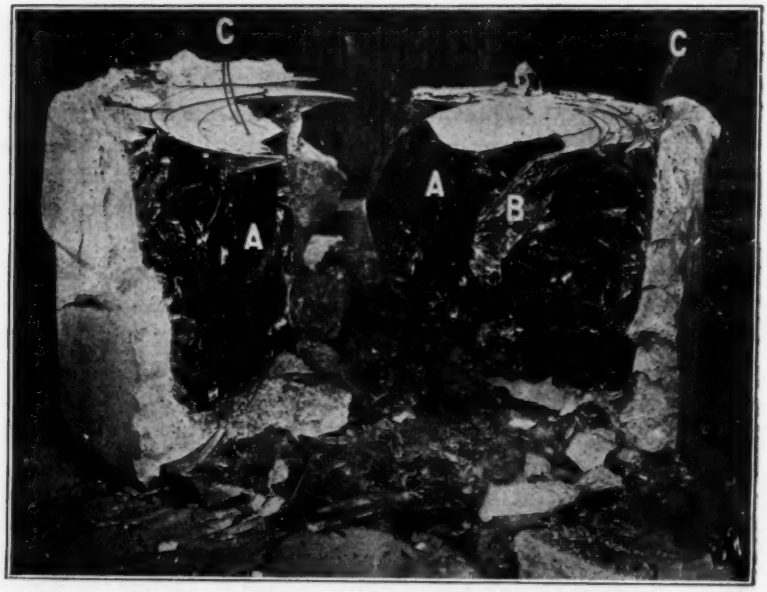

FIG. 1.—Spherical cracks in a poorly annealed melt.

that was cooled too rapidly. The cracks here are chiefly of the spherical type, appearing on the top surface as concentric circles. The inner core A (broken in the picture), was quite large, and as is usually the case, includes considerable of the top surface. The shape and irregular surface of the block B are characteristic of poorly annealed melts where the central core is not found unbroken. The wires C are thermocouples placed in the melt while it was still molten. This is an excellent example of a poorly cooled melt.

Marbles.—Where the central core remains unbroken the resulting marble is often so large as to include not only the top surface of the glass but some of the side walls of the pot as well.

When it is broken it yields blocks like B in Fig. 1; or, in extreme cases, the whole marble may be shattered by a very large number of radial cracks, as in Fig. 2. In this figure the center of the melt was at C, and S was the outside surface of the marble.

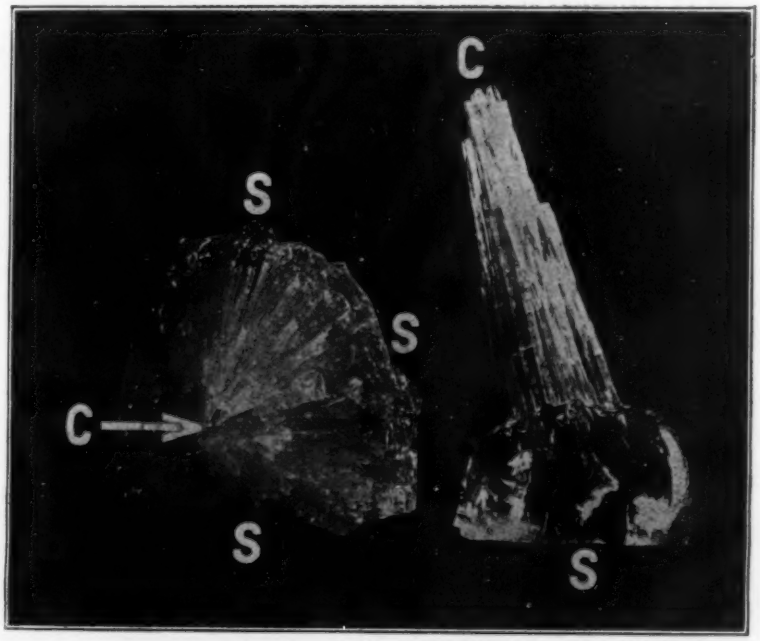

FIG. 2.—Block of glass obtained on breaking a heavily strained marble.

Plane Cracks.—When the melt has cooled to a temperature usually between 200° and 400° C, cracks start in the glass at the pot wall. These are probably due to the difference in contraction between the glass and the pot. These cracks extend into the melt until they reach another crack, or the opposite side of the pot. If the interior of the melt has not been separated from the pot by spherical cracks, the first plane crack to appear usually divides the melt in two along a vertical diametral plane. Subsequent cracks are usually not radial, but perpendicular to the first one. After a comparatively few cracks have formed,

the tendency for the cracks to follow the shortest path confines their activity to the outer portion of the melt. Here they cut the glass into pieces the size of one's fist or smaller. Horizontal cracks form as well as vertical, but are usually subordinate to the vertical cracks.

The presence of these cracks in the interior not only prevents the formation of a marble by cutting it up before it is formed but also greatly reduces the amount of strain present in the interior.

Fig. 3 is a photograph of a well annealed melt, broken in two

FIG. 3.—Plane cracks in a well-annealed melt.

along the diametral plane crack. The cracks here are entirely of the plane type, the surfaces are smooth and the blocks of glass are large. The smaller fragments are confined to the bottom and sides of the melt where the glass is certain to be full of striae dissolved from the pot.

An experiment was carried out to show the effect of the pres-

ence of plane cracks on the strain in the melt, and to prove that they were caused by the pot. A "marble" about 20 inches (50 cm.) in diameter was insulated with a thick layer of sand and heated very slowly to its annealing temperature. It was then cooled to room temperature at a rate somewhat slower than the rate at which 36-inch (90 cm.) melts were cooled. Neither spherical nor plane cracks appeared on cooling. On being cut up with a cold chisel, the marble broke into large pieces, but of irregular shape with rather rough surfaces. 36-inch (90 cm.) pots of glass, although cooled more rapidly than the above, showed plane cracks with smooth surfaces.

The temperature at which the formation of plane cracks begins seems to be nearly independent of the temperature gradient and cooling rate. Therefore it is possible, by controlling the temperature gradient, to hold back the formation of spherical cracks until the plane fracturing is well under way.

Temperature Limits of Annealing Range.—We are now in a position to define the temperature limits of the annealing range, and, from them, those of the other two stages.

If a small block of glass, placed in a polariscope, is heated rapidly, it will exhibit the characteristic colored figure of glass under strain. When the block is passing through a certain temperature range the color changes rather rapidly. We shall call the average temperature of the block at this period the "annealing temperature."[1] It depends on the size of the block and on the rate of heating, but does not vary more than 20°, provided the smallest dimension of the block is less than 1 cm. and that it is heated at a rate nearly rapid enough to crack it. The greater part of the strain present in a block of glass held at this temperature will vanish in a few seconds. Thus the extent to which strain can accumulate at this temperature is small compared with the rate at which it is introduced.

This temperature can be more definitely fixed as that at which a given proportion of the strain in a block of a certain size will disappear in a given time. For our purpose a rougher, more easily determined point is sufficient, and preferable.

[1] The "annealing temperature" determined by this method is considerably higher than the temperature usually employed in the annealing kilns.

The "annealing temperature" of the glass is here taken as the upper limit of the "annealing range" of the melt (see page 544). The lower limit is taken as the temperature at which the formation of plane cracks begins.

The lower limiting temperature depends on the relation of the coefficient of expansion of the glass to that of the pot. As a first approximation it may be assumed to be 250° to 300° below the annealing temperature.

Conditions to be Secured through Annealing Range.—As the spherical cracks are entirely due to the reduction of the temperature gradient, it is best to keep this low in the first place, and then to prevent its decreasing greatly until the formation of plane cracks is well under way. These conditions can be attained by decreasing the cooling rate at a time shortly before the annealing temperature is reached, by adding insulation or otherwise; and by removing the insulation a little while before the end of the annealing range. Removing the insulation will usually increase the temperature gradient temporarily, thereby neutralizing some of the strain present and gaining time for the plane cracks to appear.

Methods of Retarding the Cooling.

The rate of cooling through the annealing range may be retarded by placing the pot in an inclosed space, such as a kiln or pot-arch, and heating the air around it; or by surrounding the pot with a layer of insulating material. Either method gives a satisfactory result, and there seems to be no great advantage in combining them, except that in any case the surface of the glass should not be exposed, but covered by a layer of insulating material having at least the insulating properties of the pot wall itself.

Either of these methods depends on the fact that the melt tends to cool to the temperature of its surroundings at a rate that is more or less proportional to the difference between its temperature and that of its surroundings. Whether the melt is insulated by the addition of some covering or by the presence of the pot alone, the ratio of cooling rate to temperature difference decreases with increase of insulation. Thus a given rate

of cooling at any temperature may be attained by adding sufficient insulation at room temperature, or by placing the melt in a sufficiently heated enclosure, with no insulation beside that afforded by the pot and the layer of insulation previously applied to the surface of the glass.

Use of a Heated Kiln or Pot-Arch.—In this method the rate of cooling can be controlled by varying the temperature or the rate at which air is supplied to the pot. Two general methods have been developed: (1) where the temperature and rate are kept nearly constant; and (2), where the temperature and rate are decreased as the melt cools.

In Fig. 4, A, B, and C are time-temperature curves for a 36-inch

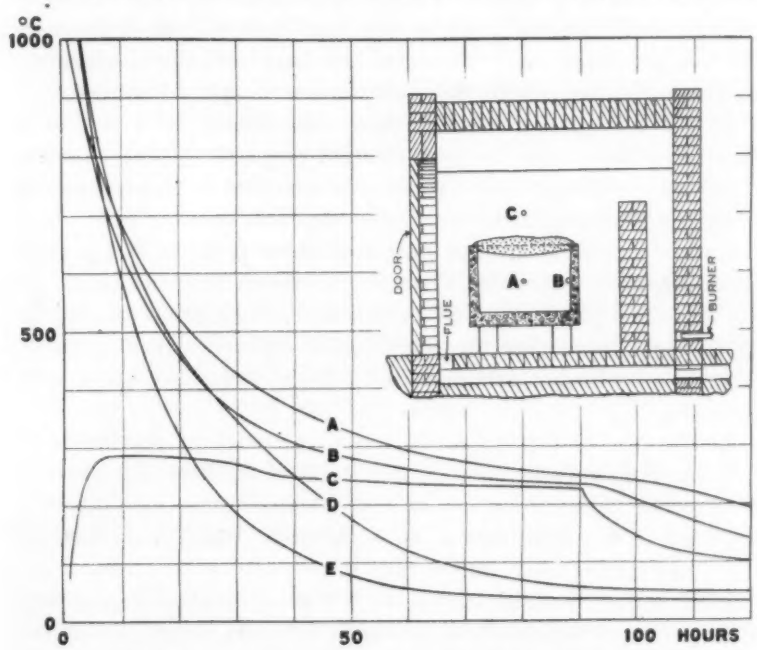

FIG. 4.—Time-temperature curves for melt cooling without added insulation. Curves A, B, and C apply to a melt cooled in a heated pot-arch; Curves D and E to a melt cooled in the open.

(90 cm.) pot of light barium crown glass, (n_D = 1.57) annealed in a heated pot-arch. Curve A represents the temperature at

the center of the melt; Curve B that at a point on the inside wall of the pot, midway between top and bottom; Curve C that of the air 8 inches (20 cm.) above the pot. The top surface of the glass was covered with a 4-inch (10 cm.) layer of kieselguhr immediately after setting the melt out of the melting furnace. About an hour later the gas was lighted in the arch. Zero time in Fig. 4 and subsequent figures is the time at which the pot was set out of the melting furnace.

Curves D and E in the same figure are for a 36-inch pot of medium flint glass ($n_D = 1.61$), insulated on the surface as before, but allowed to cool in the open at a temperature of about 30°. Curve D represents the temperature at the center of the melt and Curve E that at a point in contact with the inside of the pot wall midway between top and bottom. In both cases the temperatures were measured by bare-wire thermoelements permanently placed with their junctions at the points indicated.

As the two glasses used in these experiments have not been found to differ widely in their thermal properties, these two sets of curves are comparable and show what effect is to be expected when the temperature to which the melt can cool is raised.

Curves A and B are not only less steep than D and E, but closer together, indicating a smaller temperature difference. At the time the gas is shut off in the arch, they begin to diverge again, thus preventing the temperature difference from decreasing below its former minimum until the melt reaches a low temperature.

In the case of Curves A, B, and C, no attempt was made to cool the melt rapidly through the stage of fluidity. In practice it would be better to let it cool 3 or 4 hours in the open before it is placed in the arch; and to have the arch heated while the pot is waiting rather than after it goes in.

This method was developed with the expectation of building a long, heated tunnel through which the pots should be moved on cars; the length of the tunnel to be such that it should contain three days' run of pots under conditions of maximum production. It was found that if a slow stream of air were supplied to the tunnel at a constant temperature of 300° C, the annealing would be entirely satisfactory.

Dr. C. N. Fenner, of the Geophysical Laboratory, developed the following method at the plant of the Spencer Lens Co.: When a melt was removed from the melting furnace, it was allowed to cool freely until a new pot had been taken from an individual pot arch and set into the melting furnace. The melt, having cooled about 15 minutes in the open and until convection currents had subsided, was then placed in the hot pot-arch from which the new pot had been taken. The burners of the arch were then adjusted so that its temperature would fall to a low red heat (500 to 600°) over night (about 16 hours). He then found that the melt would anneal well: (1) if the firing were adjusted so that the temperature of the air would reach about 400° at the end of another 24 hours, or (2) if the flame were turned completely out. In the latter case, since both the firing ports and flues of the arch were close to the bottom, there was probably no tendency for air to circulate through it, and it cooled solely by the conduction of heat through the walls of the pot-arch. If, however, the flame was turned very low, but not out, a circulation of relatively cool air was induced which caused the melt to cool so rapidly that it was poorly annealed.

No data are available for the temperatures within the melt, but it seems likely that this procedure causes the temperature gradient to remain small, and to reach its maximum at a temperature well within the annealing range. Thus the strains that fracture the melt did not begin to appear until a low temperature was reached.

At the Bausch & Lomb plant, C. N. Fenner and R. E. Hall developed a method similar to that which was later used at the Spencer Lens plant, except that fire was maintained in the pot arch until it had reached a temperature of 200° to 300°. This method also probably keeps the temperature gradient low, and increases it again just after the melt has passed out of the annealing range.

These three methods are summarized in the curves of Fig. 5, which represent the temperatures of the air over or close to the pot, plotted against the elapsed time after setting the melt out of the melting furnace. The curves are in three sets. Each set consists of a solid, a dashed, and a dotted curve, showing the

temperature regulation which resulted in good, fair, and poor annealing, respectively, for each of the three methods described below (Pittsburgh, Spencer, and Bausch & Lomb). These curves were made on various types of glass and sizes of pots, and the character of the annealing was judged by three different men at three different plants. Thus the three sets cannot be directly compared.

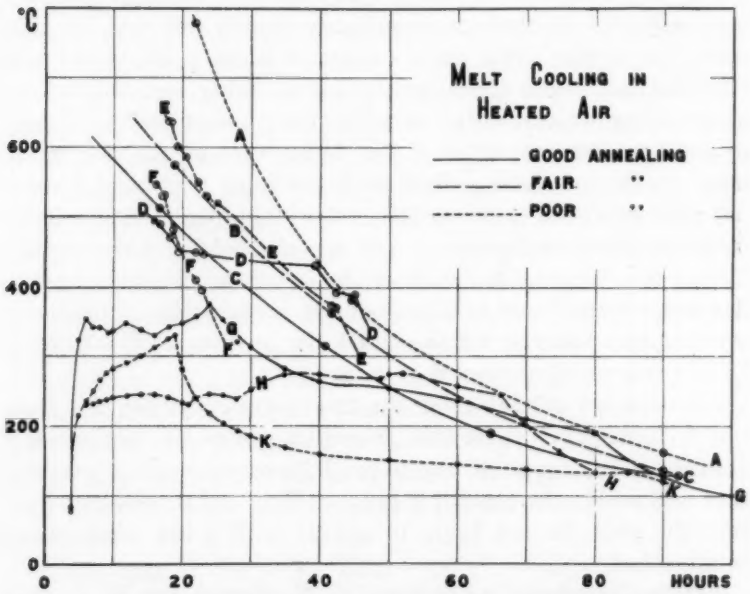

FIG. 5.—Time-temperature curves for melt cooling in heated air. The cooling was retarded by placing the pot in a heated pot-arch. The temperatures are those of the air over, and close to the pot.

Curves A, B, and C are plotted from data on the temperature of the air above the pot, taken at the Bausch & Lomb plant, where the fire was kept burning in the arch until it had cooled to a low temperature. Each of these three curves was plotted from 3 (in one case 4) measurements, which accounts for their marked regularity. In spite of the fact that the lowest average cooling rate during the first three days was in the case of A, nevertheless A gave the poorest annealing. Here the arch was

kept for a long time at a high temperature, which greatly reduced the temperature gradient at this temperature, but did not prevent its reaching a high value as the melt passed rapidly through the annealing range. In the case of B and C, the melt cooled rapidly at high temperatures, but the cooling was greatly retarded through the annealing range.

Curves D, E, and F represent data taken at the Spencer Lens plant where the gas was left burning until the arch had cooled to about 400°. This set also illustrates the fact that early retardation is not important in securing satisfactory annealing.

The data for Curves G, H, and K were taken at the Charleroi plant of the Pittsburgh Plate Glass Co.; they are part of the same series of experiments as Curves A, B, and C of Fig. 4. In the case of G, while the annealing was entirely satisfactory, it was found later that the rate of cooling was slower than necessary. In the case of H, a somewhat slower rate seemed desirable at the start. Where the curve was more nearly the mean of G and H, it was found that a 36-inch melt could be removed from the arch at the end of $2^1/_2$ days, and broken at the end of 4 days. Curve K resulted, as was expected, in a marble. This schedule was designed to apply alike to all of the several types of glass made at that plant, and could therefore be somewhat shortened for individual cases.

Use of Insulation around the Melt.—Insulation may be applied to the pot either in the form of an insulating cover, or the pot may be surrounded by a layer of some loose insulating material such as sand or kieselguhr. Where sand is used, a thickness of 8 inches (20 cm.) is sufficient for pots 30 to 50 inches (75 to 120 cm.) in diameter. This may be reduced, in the case of kieselguhr, to 3 inches ($7^1/_2$ cm.).

The form of cover used at the Pittsburgh Plate Glass Company's plant consisted of a hollow double-walled sheet iron cylinder, closed at one end by a double wall. The 3-inch ($7^1/_2$ cm.) space between the walls was filled with kieselguhr, and two handles were riveted to the outside to facilitate handling. The height inside the cover was such that, when it was inverted over a pot, the bottom edge of the cover rested on the floor, and could be

sealed with a little sand. The weight of such a cover for a 36-inch pot (90 cm.) was perhaps 150 lb. (68 kg.).

At the same plant, previous to the use of these covers, the melts were insulated with sand, held in place by a retaining case of sheet iron. These cases were made by riveting together two sheets of flat galvanized iron to form a continuous band. Their average diameter was greater than that of the pot, by 16 inches (40 cm.) or more, and their height was 30 inches (75 cm.). This height brought the top of the case to about the same level as the surface of the glass in the pot.

These cases were filled by shoveling the sand into them, an operation that consumed considerable time, as it involved moving about a ton of sand for each 36-inch pot. When the sand was to be removed from the pot, the case was first lifted off by the crane, allowing the sand to form a cone around the bottom of the pot. This cone was then hoed back to clear the pot.

Of the two, the portable covers were preferable, entailing less time and labor and being more tidy. They were objected to at first on the ground that circulation in the air space between the pot and the cover might induce too rapid cooling. This was found not to be the case.

In Fig. 6 time-temperature curves are plotted for the cooling of two melts of medium flint glass ($n_D = 1.61$) in 36-inch (90 cm.) pots. In the case of Curves A, B, and C, the melt cooled freely for 5 hours after it was set out of the furnace; it was then insulated with a 10-inch (25 cm.) layer of sand retained by a sheet-iron case; this insulation was removed 62 hours after setting out. Curve A represents the temperature at the center of the melt; Curve B, that at a point in contact with the inside of the pot midway between top and bottom; and Curve C, that on the outside of the pot opposite point B. The melt, sand and retaining case are drawn to scale in the upper part of the figure, and the location of the thermocouples shown by small circles. In the case of Curves D and E, the melt was cooled in the open with no insulation other than a 4-inch (10 cm.) layer of kieselguhr on top. Curve D is for a point in the melt corresponding to that of Curve A; Curve E, for a point corresponding to that of Curve E. These two curves are the Curves D and E of Fig. 4.

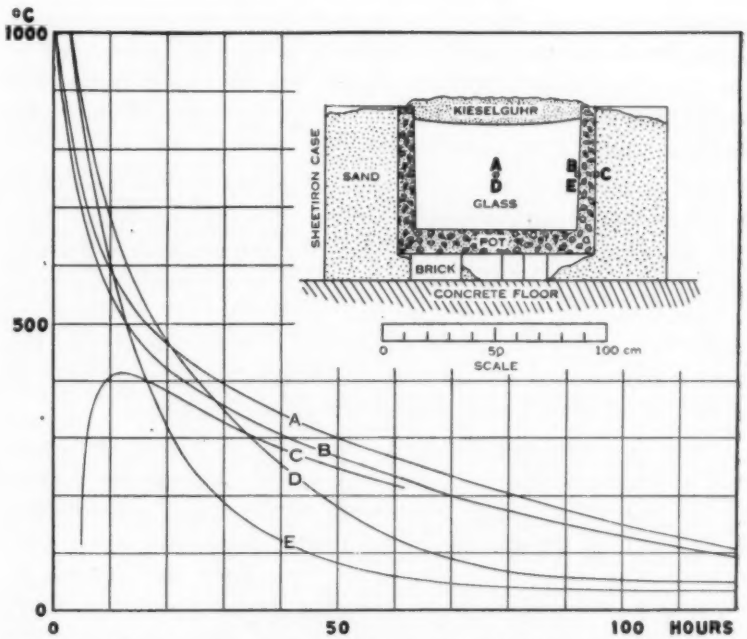

FIG. 6.—Time-temperature curves for a melt cooled in sand. Curves D and E of Fig. 4 are reproduced for comparison.

Course of the Temperature Drop.—In Fig. 7 the difference in temperature between the center and side of the melt, for the three

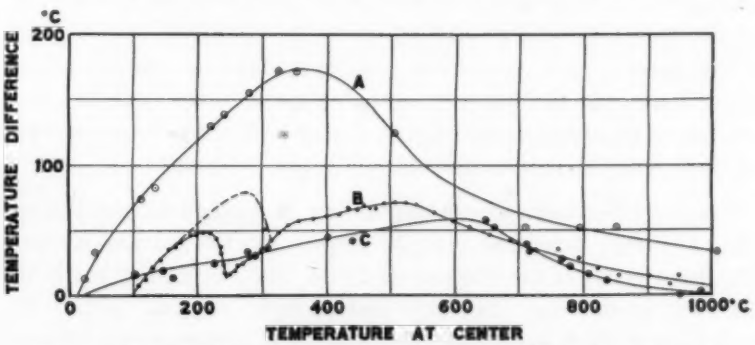

FIG. 7.—Temperature drop through the melt plotted against the corresponding temperature at the center, for the three melts of Fig. 4 and Fig. 6.

melts of Fig. 4 and Fig. 6, are plotted against the corresponding temperatures at the center. Curve A is for the melt cooled in the open (D and E of both Fig. 4 and Fig. 6); Curve B, for that cooled in a pot arch (A, B, and C of Fig. 4); Curve C, for that cooled in sand (A, B, and C of Fig. 6).

Most of the cracks in melt A were of the plane type, and there were many fragments weighing 10 lbs. ($4^1/_2$ kg.) or over. This may be explained by the fact that the temperature difference did not begin to increase until the outside of the melt had cooled to about 200°, a temperature well below that at which plane fracturing usually begins in this type of melt. However, the break could not be considered as satisfactory because of the large proportion of glass found broken into small fragments.

Curve B shows two maxima. The first, at about 510°, was only attained after the center had cooled through more than 500° at high temperatures; the second, at about 210°, was reached on shutting off the gas in the arch after the center had cooled through only 40°. This shows the very great role played by convection and radiation in reducing the temperature drop. The portion of the curve to the left of 250° gives an idea of the extent to which the fall of the temperature drop can be postponed. In later experiments the gas was turned out in the arch much sooner, which would have caused the curve in this case to take a form something like that indicated by the dotted line in the figure. This practice resulted in as good a break and a very much shortened time of cooling.

The lower portion, below 260°, of Curve C shows a much smaller break than that of Curve B, probably because the cone of sand was not completely removed from that side of the pot—an experimental detail that was unfortunately not recorded.

Cooling Schedule, where Insulation is Applied to the Pot.—The following schedule was developed by the author at the Charleroi plant of the Pittsburgh Plate Glass Company for cooling their 36-inch (90 cm.) pots and 49-inch (125 cm.) pots. As each type of glass was set out of the melting furnace at its definite temperature, the operations of cooling were performed according to the time that had elapsed after the pot was set out. The

schedule was so arranged that all pots of the same size were treated alike, regardless of the type of glass they contained.

Immediately on setting the melt out of the furnace, the surface of the glass was covered with a 4-inch (10 cm.) layer of kieselguhr and the pot placed on three bricks to cool.

Four hours later in the case of 36-inch pots, and eight hours in the case of 49-inch pots, insulation was applied to the pot in the form of an 8-inch (20 cm.) layer of loose sand or in the form of one of the covers described above.

The insulation was removed from the 36-inch pots 3 days after setting out; and from the 49-inch pots 5 days after setting out. Two or three days later the melts were cool enough to be broken down.

Where 36-inch melts are cooled in sand according to this schedule the very approximate temperatures given in Table 1 may be expected at the center of the melt. The third and fifth columns of numbers refer to the times insulation is applied and removed, respectively. Some of the annealing temperatures given in the fourth column were determined by G. W. Morey, of the Geophysical Laboratory, using the rough method given above; the others were extrapolated from data obtained for the release of strain at lower temperatures by L. H. Adams and E. D. Williamson, of this laboratory.

TABLE 1.

Approximate Temperatures at Center of 36-inch Melts Cooled in Sand.

Type of glass.	n_D.	Temp. set out.	Temp. after 4 hrs.	Annealing temp.	Temp. after 72 hrs.
Flint.....	1.57	1038°	800°	465°	320°
Flint.....	1.61	996	780	455	300
Flint.....	1.66	968	760	445	290
Crown, Ordinary.....	1.52	1093	850	570	350
Crown, Borosilicate....	1.52	1116	860	590	360
Crown, Barium.....	1.57	1016	790	580	310
Crown, Barium.....	1.61	1016	790	600	310

Breaking Down.—When the melt is ready to be broken down, the diametral crack in the glass is first located, and the pot broken

at the ends of it with a sledge. The two halves of the melt are now separated and the blocks of glass loosened by beating on the outside of the pot, or shaking it with a crow bar. A small hammer is used where necessary to free the blocks of adhering pot shell.

Summary.

The conditions to be attained when a melt of optical glass is cooled in the pot are: (1) that neither ream nor bubbles shall be introduced during the cooling, nor carried into the middle of the melt; (2) that the glass shall not become inhomogeneous through the precipitation of a crystalline phase; (3) that the bulk of the cooled melt shall be found cracked into large, reasonably rectangular blocks, having smooth, flat surfaces; and (4) that these blocks shall be sufficiently free from strain to cleave readily with a smooth fracture.

The appearance of ream in the middle of the melt, vacuum bubbles and a crystalline phase, can be discouraged by rapid cooling, preferably from the bottom of the pot, while the glass is still soft; and by insulating its top surface as soon as the melt is set out of the melting furnace.

The cold melt shows cracks of two types: "spherical cracks," and "plane cracks." The spherical cracks are due to decrease of temperature difference, and first appear below the temperature at which the drop is a maximum; these cracks form one after another until the temperature gradient has disappeared. The plane cracks seem to be the result of a difference in rate of contraction between the glass and the pot; the temperature at which they first appear in a given melt seems to be nearly independent of the cooling rate. The presence of either type of crack in the melt reduces the tendency of the other type of crack to form. As the fracture due to spherical cracks is rough and the pieces formed are irregular, while that due to plane cracks is entirely satisfactory, it is desirable (1) to maintain the temperature gradient at a low value by slow cooling; and (2) to prevent its decreasing much below its maximum until after the formation of plane cracks has begun, by increasing the cooling rate at the proper time.

The cooling rate can be reduced (1) by heating the surroundings of the melt, *i. e.*, placing it in a heated kiln; and (2) by surrounding it with an envelop of some such insulating material as sand, kieselguhr, etc. Variations of these two methods are described and time-temperature data given.

GEOPHYSICAL LABORATORY,
CARNEGIE INSTITUTION OF WASHINGTON,
April, 1919.

SPECIAL SPARK-PLUG PORCELAINS.[1]

By A. V. Bleininger and F. H. Riddle.

Requisite Properties of Porcelains for Spark Plugs.—The average commercial porcelain does not fulfill the conditions required for spark-plug service, nor for any other conditions where high-tension currents are employed and the temperature is considerably above atmospheric conditions. From the standpoint of the electrical resistance at the temperatures reached in airplane engines, the feldspathic porcelains begin to break down at a rate rapidly increasing with temperature, due to electrolytic effects. This is indicated also by the polarization which is observed in using direct current. The leakage thus taking place may become a serious factor.

The average porcelain is likewise not constant in volume and the volumetric changes are greater than can be explained solely by thermal expansion. Aside from the thermal expansion, the quartz content of porcelain, when heated or cooled, is subject to certain modifications in crystalline structure, accompanied by definite volume changes. Thus, the form of quartz permanent at atmospheric temperature, is alpha quartz which inverts to beta quartz at 570° C, a transformation which is reversed by cooling. On the other hand, if, in the firing of porcelain, the quartz is inverted to its high-temperature form, cristobalite, there is again the transformation of the alpha to the beta modification to be considered, which takes place at 230° C. In either case, volume changes are unavoidable in all porcelains containing free quartz in large amounts, due to the inversion noted. In a good spark-plug porcelain, the quartz should be eliminated from the composition and replaced by a substance not subject to these inversions.

Another defect of the ordinary porcelain is that the coefficient of thermal expansion is by no means constant at different tem

[1] By permission of the Director, Bureau of Standards.

peratures. It may be 19 × 10^{-6} between 30–200° C and 5 × 10^{-6} between 400–500° C.

With respect to mechanical strength, also, great variations are possible. A series of tests of commercial electrical porcelains, conducted at the Pittsburgh laboratory of the Bureau of Standards, showed variations in the modulus of elasticity of from 1,600,000 to 6,000,000.

Methods of Testing Spark-Plug Bodies.—For the purpose of studying porcelains from the standpoint of their electrical conductivity at the high temperatures obtained in airplane ignition systems, a large number of typical compositions were made up in the form of test cups of 60 mm. outside diameter and 65 mm. high, the thickness being 2.5 mm. These mixtures were usually prepared in 10 kg. batches, carefully weighed out and ground wet in porcelain-lined ball mills for three hours. The suspension of clay was pumped into a filter press to remove the water and then kneaded in a mixing machine to the consistency required for shaping the mass on the potters wheel. This was done by the use of plaster molds in which the cups were "jiggered" in the usual manner. The specimens were then dried, placed in saggers and burned in a down-draft kiln fired with natural gas to the finishing temperature. The latter was controlled both by means of thermocouples with the necessary galvanometers, and pyrometric cones. Each cup was examined for non-absorption by the application of ink. The testing of the specimens was done at the Washington laboratory. The characteristic expression for the resistivity of the porcelains is the "Te" value, which represents the temperature in degrees C at which a cubic centimeter of the material still shows a resistance of one megohm.[1]

Replacement of Feldspar.—Upon comparing the "Te" value of the different porcelains it was noted that there exists no definite relation between the composition and the electrical conductivity. On the other hand, the higher the maturing temperatures of the porcelains, the higher was the "Te" value. This is practically equivalent to saying that, roughly speaking, the electrical resistance at higher temperatures is the greater, the lower the feldspar content, since small amounts of this flux make it neces-

[1] *J. Am. Ceram. Soc.*, 1, 700 (1918).

sary to carry the porcelain to a higher burning temperature. The relation, however, is not sharply defined, as may be observed from the results compiled in the following table:

TABLE 1.

No. of body.	Per cent feldspar.	Maturing temperature in cones.	"Te" value.
16.....	16	16	560
17.....	18	15	390
18.....	20	14	440
22.....	28	14 ($^1/_2$ over)	370
23.....	30	13	450

It is quite evident that the micro-structure of the porcelains is an important factor in this connection, since it cannot be immaterial how much kaolin or quartz has been dissolved by the fused feldspar and how much sillimanite has been formed. The evidence, however, was considered sufficient to warrant the replacement of the feldspars by other fluxes, the oxides of magnesium and beryllium being used for this purpose.

Owing to the evolution of carbon dioxide from magnesite and the artificially prepared basic magnesium carbonate during the firing process, and the very large shrinkage accompanying their use as a flux, it was decided to introduce the magnesia in the form of a calcine; that is, a silicate mixture previously fired tovitrification. The mixtures employed for this purpose corresponded to the formulas: $MgO.Al_2O_3.2SiO_2$; $MgO.Al_2O_3.4SiO_2$ and $MgO.Al_2O_3.6SiO_2$. Of these, the first and the second were most used in this work. The preparation of the calcines consisted in dry ball-mill-grinding and firing the mixtures made up into balls with just sufficient water. After calcination, the material was crushed and ground and introduced into the bodies in this form. The beryllium oxide was brought in through the use of the mineral beryl (which has the general composition $3BeO.Al_2O_3.6SiO_2$) without any previous heat treatment.

With the use of magnesia as the principal flux the electrical resistance, and hence the value "Te," was found to increase quite decidedly, though with no well defined regularity as referred to

percentage content of magnesia. It was seen again, in this connection, that the structure of the porcelain plays an important part especially as it is known that MgO accelerates the crystallization of sillimanite most vigorously. Not only the number but also the size of the sillimanite crystals is of significance in determining the texture of the body, whether it is to be fine-grained, glassy, or coarsely crystalline. The rate of cooling the porcelain is likewise of importance since a rapid drop in temperature invariably causes the structure to be closer and of more vitreous character than when a longer time is taken in cooling down the kiln. For this reason smaller kilns are to be preferred to larger ones since they permit of more rapid cooling. The effect of magnesia, added in the form of synthetic silicate, is strikingly shown by the high "Te" values of bodies Nos. 77 and 78 (see Table 4).

It is a fact that the magnesian silicates show electrolytic effects which are much less prominent than when feldspar is used as a flux. It likewise appears that the higher the firing temperature of the porcelain the greater its electrical resistance, at temperatures up to 700° C, or somewhat above this point.

With reference to the use of beryllium oxide, it was found that this flux behaves similarly to the magnesia in showing high electrical resistance and "Te" values. This is indicated by the following table:

TABLE 2.

No.	Per cent beryl.	Per cent clay.	Per cent flint.	Maturing temp. of porcelain in cones.	"Te" value.
193....	25	50	25	12	624
194.....	35	50	15	11	784
195.....	45	50	5	11	798

It is evident from these results that beryllium oxide, used in the form of the mineral beryl, is a valuable flux from the standpoint here under consideration. It requires, however, careful temperature control in firing, since the beryllium porcelain is subject to sudden deformation as the vitrification temperature is exceeded. The experiments have proven that beryllium oxide

is worthy of consideration for the production of such porcelain, and the high "Te" value obtained, 798, is exceedingly promising. The firing temperature of these bodies is quite low, from cones 11 to 12.

Another interesting fact was that the thermal expansion of the beryllium porcelain was found to be lower than that of the feldspathic bodies commonly employed. The average coefficient was found to be 1.63×10^{-6} for the temperature interval 26° to 200° C; 2.95×10^{-6} for 200° to 400°; 3.60×10^{-6} for 400° to 570°; and 2.33×10^{-6} for 26° to 400°.

Replacement of Quartz.—From a general study of porcelains, it appears desirable to eliminate the quartz, as has been pointed out in a previous paragraph. Some of the materials available for this purpose are calcined kaolin, synthetically prepared sillimanite ($Al_2O_3.SiO_2$), sintered or fused alumina, and zirconium oxide. These substances have been introduced in a number of compositions. The effect of adding calcined kaolin in general was beneficial; even with a feldspar content of 13.5 per cent (body No. 63) a fair "Te" value, 540, was obtained. This particular composition contained 50 per cent raw kaolin, 13.5 per cent feldspar, 1.5 per cent calcium carbonate, and 35 per cent calcined kaolin. The maturing temperature of this porcelain was that corresponding to cone 16. It is evident that by raising the content of calcined kaolin still more at the expense of the feldspar a higher "Te" value would be obtained. The introduction of plastic ball clay to replace kaolin, invariably and in all types of bodies, reduces the electrical resistance within the temperature range here under consideration.

The use of fused alumina in any extensive work was prohibited by the lack of material low in iron content. The commercial substance is unsuited for this purpose. The total amount of white, fused alumina available was not more than two pounds. To bring about the necessary impervious and dense structure the use of 17.6 per cent of feldspar was required. A mixture consisting of 45 per cent kaolin, 17.6 per cent feldspar, 4.4 per cent calcine No. 13, and 33 per cent fused alumina, resulted in a very tough porcelain which showed a "Te" value of 620. Cal-

cine No. 13 was compounded, according to the formula $MgO.Al_2O_3.2SiO_2$, from 84 parts, by weight, of magnesium carbonate, and 258 parts of plastic kaolin from Florida and Georgia. Here again the reduction of the feldspar content and its replacement by calcine No. 13, or beryl, or a combination of these two, would be certain to raise the "Te" value considerably and at the same time would result in a porcelain of excellent mechanical properties. The cost of the fused, white alumina would be quite high, but would be justified under the circumstances. Furthermore, a single calcine could be readily produced by combining the raw materials of calcine No. 13, the kaolin and magnesite, with uncalcined alumina from any convenient source, and firing the mixture to a point of constant volume, which could be accomplished at temperatures not exceeding cone 18. At the same time this procedure would simplify the process of preparation very considerably.

Sillimanite ($Al_2O_3.SiO_2$) is a normal component of all hard-fired porcelains, and this constituent, if not present in the form of large crystals, imparts to the material constancy in volume upon heating, lowers the thermal expansion, and increases the refractoriness and the resistance to sudden heating and cooling. For this reason it was thought desirable to produce the mineral synthetically and to introduce it in the porcelain composition in place of the quartz. This was done by combining 70.2 per cent of plastic kaolin, 27.8 per cent anhydrous alumina, and 2 per cent boric acid. After grinding this mixture in the dry state in a ball mill, it was made up with water to form a plastic mass which was molded into balls which were calcined to a temperature corresponding to that of cone 20, or approximately 1530° C. At this temperature the mass sintered to a dense structure, subject to but little additional shrinkage and containing only a small amount of uncombined alumina. The boric acid was added to assist in bringing about the necessary shrinkage and closing up of the pores, and it is quite probable that most of this constituent is volatilized at the final temperature of calcination. This calcine (No. 19) was then crushed separately, passed over a magnetic separator and then ground together with the other materials of the body with the addition of water, in the ball mill. In preparing

this type of body the feldspar was eliminated, on the basis of the results discussed above, and replaced by a calcine corresponding to the formula $MgO.Al_2O_3.4SiO_2$ (No. 14). This porcelain (No. 152) was composed of 30 per cent kaolin, 10 per cent ball clay, 40 per cent sillimanite calcine and 20 per cent fluxing calcine No. 14, and fired to a temperature of cone 16 or higher. The resultant material had excellent electrical qualities and an average "Te" value of 690. The structure of this body should be fine and dense, a condition which requires quite rapid cooling of the kiln. Slower cooling results in the formation of coarser sillimanite crystals which produce a structure much less resistant to sudden heating and cooling.

The preparation of this body may be simplified by the combination of the two calcines into one. Thus, by employing the composition of flux No. 13 ($MgO.Al_2O_3.2SiO_2$) the combined calcine would be composed of 75.10 per cent plastic kaolin, 8.77 magnesite, and 16.13 per cent anhydrous alumina. The boric acid content has been eliminated. Body No. 152 would then consist of 30 per cent kaolin, 10 per cent ball clay, and 60 per cent of the combined calcine. Wherever the working conditions permit, the ball clay should be eliminated, and an effort made to employ the body containing 40 per cent of plastic kaolin, which can usually be done by allowing the mixture to age before molding it.

The tendency of the body to crystallize may be diminished also, by replacing the fluxing calcine in part by beryl, so that these two components are present in the ratio of 1 to 1. If, then, the combined fluxing- and sillimanite-calcine is employed, the resultant composition would be: kaolin 60.94 per cent, anhydrous alumina 18.38 per cent, boric acid 1.32 per cent, magnesite 4.58 per cent, and beryl 14.78 per cent. The body, as before, would consist of 60 per cent of this calcine and 40 per cent of kaolin. The fusion point of this mixture is so low that it would be quite possible to eliminate the boric acid entirely. At the same time the vitrification point of the body is lower and closer to the normal kiln-temperatures.

Attention might be called to the fact that the use of a silicious porcelain is not objectionable from the electrical standpoint, but only from consideration of the mechanical strength, resistance

to sudden temperature changes, etc. This is shown by the results upon high silica porcelains, bodies Nos. 116 and 119, which show a very high "Te" value, namely 730. This type of porcelain contains 50 per cent clay, 30 per cent free quartz (flint) and 20 per cent magnesia calcine (Table 3). In No. 116, the calcine corresponds to the formula $MgO.Al_2O_3.2SiO_2$, and in No. 119 to $MgO.Al_2O_3.4SiO_2$. It is fair to state that these porcelains would give excellent results, considered from the dielectric standpoint, when used under conditions not so extreme with reference to temperature and shock—as is the case with spark plugs used in airplane engines.

Zirconium oxide was used only in two porcelains, due to the comparatively limited supply available. The material at hand was zircon which contained 52.74 per cent of ZrO_2 and 43.46 per cent of silica. As received, the mineral was high in iron, which, however, was eliminated by treatment with chlorine[1] at a temperature of 800° C. In this manner all but a trace of the iron was removed. Owing to the fact that these zircon porcelains were produced comparatively early in the work, feldspar was employed as the flux. The result was that the electrical resistance, and hence the "Te"

TABLE 3.

Body No.	Per cent clay.	Per cent quartz (flint.)	Per cent MgO-calcine.	Formula of MgO-calcine.	Sillimanite calcine, per cent.	Coefficient of thermal expansion x 10^{-6}. Degrees centigrade.			
						30–200°	200–400°.	400–550°.	30–400°.
114	50	20	30	$MgO.Al_2O_3$-$6SiO_2$	..	19.45	9.35	5.52	13.99
116	50	30	20	$MgO.Al_2O_3$-$2SiO_2$	..	7.34	6.11	4.68	6.68
117	50	20	30	$MgO.Al_2O_3$-$2SiO_2$	..	8.93	4.43	4.05	6.51
119	50	30	20	$MgO.Al_2O_3$-$4SiO_2$	..	19.61	11.13	8.08	15.03
120	50	20	30	$MgO.Al_2O_3$-$4SiO_2$	..	10.45	5.43	4.45	7.74
152	40	..	20	$MgO.Al_2O_3$-$4SiO_2$	40	3.36	4.19	4.78	3.81

[1] A. J. Phillips, *J. Am. Ceram. Soc.*, 1, 791 (1918).

value, was low, being 500 and 450, respectively. With the use of the magnesia- or beryllium-fluxes much better products could undoubtedly be produced. The mechanical strength of these porcelains was excellent in every respect. The composition of these bodies, Nos. 72 and 73, is given in Table 4.

The effect of the quartz in porcelain upon the thermal expansion of the body is shown in the results compiled in Table 3.

TABLE 4.

Number of body.	16	17	18	22	23	24	28	32	35	36	39	40
	%.	%.	%.	%.	%.	%.	%.	%.	%.	%.	%.	%.
Kaolin	45.00	45.00	45.00	45.00	45.00	50.00	50.00	55.00	55.00	55.00	55.00	60.00
Feldspar	16.00	18.00	20.00	28.00	30.00	16.00	24.00	14.50	20.50	22.50	28.50	16.00
Potters flint	39.00	37.00	35.00	27.00	25.00	34.00	26.00	29.00	23.00	21.00	15.00	24.00
Whiting-($CaCO_3$)	...	...	...	...	...	...	...	1.50	1.50	1.50	1.50	...
Ball clay	...	...	...	...	...	...	...	...	...	...	...	...
Calcined-kaolin	...	...	...	...	...	...	...	...	...	...	...	...
Calcine No. 3	...	...	...	...	...	...	...	...	...	...	...	...
Calcine No. 19	...	...	...	...	...	...	...	...	...	...	...	...
Calcine No. 20	...	...	...	...	...	...	...	...	...	...	...	...
Zircon	...	...	...	...	...	...	...	...	...	...	...	...
Maturing temperature in cones	16	15	14	14	13	15	14	14	12	12	12	13
"Te" value.	560	390	440	370	450	380	358	400	460	400	390	410

TABLE 4 (*Continued*).

47	48	49	50	55	63	70	72	73	74	77	78	79
%.	%.	%.	%.	%.	%.	%.	%.	%.	%.	%.	%.	%.
60.00	50.00	50.00	51.00	50.00	50.00	50.00	37.00	37.00	....	5.00	10.00	10.00
30.00	24.00	22.00	25.00	28.50	13.50	16.40	22.00	17.60	....	....	....	....
10.00	5.00	12.00	...	...	....	....	....	....	....	....	....	....
...	1.00	1.00	1.50	1.50	1.50	1.00	....	....	....	....	....	....
...	...	...	...	...	....	....	5.00	5.00	15.00	10.00	10.00	15.00
...	20.00	15.00	...	20.00	35.00	....	....	....	....	....	....	....
...	...	...	...	...	....	4.10	....	4.40	....	....	....	....
...	...	...	22.50	...	....	28.50	....	....	....	....	....	....
...	...	...	...	...	....	....	....	....	85.00	85.00	30.00	75.00
...	...	...	...	...	....	....	36.00	36.00	....	....	....	....
10	14	14	14	14	16	14	13	13	13	13	13	13
400	410	400	390	400	540	460	500	450	640	800	790	640

TABLE 5.

Number of body.	88	94	95	107	109	116	152	180	193	194	195
	%	%	%	%	%	%	%	%	%	%	%
Kaolin.....	50.00	45.00	40.00	47.00	25.00	50.00	30.00	69.00	40.00	40.00	40.00
Feldspar.....	8.00	17.60	....	14.00	....	....	....	....	....	....	....
Potters' flint....	30.00	....	42.00	21.00	....	30.00	....	5.75	25.00	15.00	5.00
Mag. carbonate	....	....	....	10.00	....	....	....	4.58	....	....	....
Ball clay.....	....	....	....	8.00	20.00	....	10.00	9.15	10.00	10.00	10.00
Calcined kaolin	....	....	....	....	35.00	....	....	....	....	....	....
Calcine No. 8*a*	....	....	....	....	20.00	....	....	....	....	....	....
Calcine No. 13	12.00	4.40	....	....	....	20.00	....	....	....	....	....
Calcine No. 14	....	....	18.00	....	....	....	20.00	....	....	....	....
Calcine No. 19	....	....	....	....	....	....	40.00	....	....	....	....
Calcine No. 20	....	....	....	....	....	....	....	....	....	....	....
Calc. Al_2O_3....	....	33.00	....	....	....	....	....	11.52	....	....	....
Beryl.....	....	....	....	....	....	....	....	....	25.00	35.00	45.00
Maturing temperature in cones.....	15	16	20	12	16	18	16	20	12	11	11
"Te" value....	590	620	630	480	610	730	690	640	624	784	798

TABLE 6.—COMPOSITIONS OF CALCINES.

Calcine number.	$MgCO_3$. %	Kaolin %	Flint %	Calc. Al_2O_3. %	Boric acid. %	Calcination temp. (cones).
3.....	39.50	60.50	...	...	...	10
8a.....	14.40	44.30	41.30	...	...	12
13.....	23.85	76.15	...	...	...	12
14.....	18.20	56.00	25.80	...	...	13
19.....	...	70.20	...	27.80	2.00	20
20.....	34.30	...	65.70	...	...	16

It is at once evident from a comparison of these figures that the thermal expansion of these magnesia porcelains increases with the silica content of the body, and at the same time, is subject to decided variation within the temperature limits of 30–550° C. On the other hand, the porcelain in which the clay is lower, and all the quartz has been replaced by sillimanite, shows both the lowest thermal expansion and the least variation in the value of the coefficient. However, the composition is not the only determining factor and the importance of the micro-structure must be realized.

Summary.

The work, the results of which have been given, has been successful in showing: first, the injurious quality imparted to electrical porcelains by the use of feldspar as a flux; and second, in bringing out the desirability of replacing the quartz by minerals, or synthetically prepared materials, which are more constant in volume when heated. The remedial procedures advocated are, hence: (1) The replacement of feldspar by other fluxes, such as silicates of the type $MgO.Al_2O_3.2SiO_2$, or $MgO.Al_2O_3.4SiO_2$, or other silicates of beryllium and the alkaline earths, either natural or prepared artificially. (2) The elimination of quartz and the substitution of substances not subject to inversions or other volume changes. These may be highly calcined kaolin, alumina, zirconia or sillimanite, either natural or produced synthetically.

The compositions of the most typical porcelains produced in this work are compiled in Tables 4, 5 and 6, in which the "Te"

value is given for each body, so that these tabulations may be consulted for detailed information.

All of the electrical measurements as well as the determinations of the thermal expansions of the porcelains have been made in the Washington laboratories of the Bureau of Standards.

BUREAU OF STANDARDS,
PITTSBURGH, PA.

DISCUSSION OF AN ARTICLE BY N. L. BOWEN ENTITLED "DEVITRIFICATION OF GLASS"[1] IN THE APRIL NUMBER OF THE JOURNAL OF THE AMERICAN CERAMIC SOCIETY.[1]

S. F. Cox: The able discussion of the question of devitrification of glass by N. L. Bowen recalls some work done on that subject by A. E. Williams and the writer.[2] In a study of the effect of barium salts on the physical properties of glasses, the effect of increasing barium content on the devitrification tendency was noted.

Starting with a glass of the formula $\left.\begin{matrix} 0.5\ Na_2O \\ 0.1\ ZnO \\ 0.4\ K_2O \end{matrix}\right\}$ 2.5 SiO_2,

barium oxide was used to progressively replace soda, 0.1 mol. BaO replacing an equivalent amount of Na_2O, until all the latter had been eliminated; the zinc and potash were maintained constant throughout. A series would hence consist of six glasses of the same silica content. Seven such series of glasses were made, the silica content increasing by 0.5 mol. in each successive series. The last glass of the series would hence have the following composition:

$$\left.\begin{matrix} 0.5\ BaO \\ 0.1\ ZnO \\ 0.4\ K_2O \end{matrix}\right\} 5.5\ SiO_2$$

In the study of devitrification tendency, the method followed was similar to that used by Bowen. Definite heating curves were followed, the temperature being raised on successive heatings until a point well past the deformation point of most of the glasses had been attained. Fifteen of the forty-two glasses, those in the high-silica low-barium corner of the diagram, showed at 700°

[1] *J. Am. Ceram. Soc.*, **2**, 261 (1919).

[2] *Trans. Am. Ceram. Soc.*, **18**, 315–342 (1916).

C surface devitrification which was increased upon further heating at 800° C. All other glasses were entirely free from devitrification —those glasses which showed it did so only on the surface.

The tendency to devitrify at the surface of the glass is mentioned by Bowen,[1] who states that the cause is unknown. We also made note of this tendency, but were inclined to accept the explanation of Brockbank,[2] who attributes it to the volatilization of the alkali oxides from the surface, leaving a surface layer higher in silica. If the glass were nearly saturated with respect to this component, there would be a tendency for it to deposit on the surface. There would be no tendency to establish equilibrium with the glass in the interior of the piece, because of the high viscosity of the medium at these temperatures. The effect would hence be confined to the surface.

As before stated, high silica seemed more likely to produce devitrification than increasing barium content. Those glasses higher in soda, low in barium, and high in silica, tended most readily to devitrification. There was no observable connection between this property and any of the other physical properties studied.

In the light of Bowen's observations, we are inclined to believe that the reason so little devitrification was experienced in the glasses studied by us lies, among other considerations, in their high potash content and in the fact that no arsenic was used in the batches. We believe surface devitrification to have been due to the high silica content, particularly at the surface, as it occurred in those glasses highest in silica. Identification of this surface material, which was little more than a strongly adhering film, was not possible. Tendency towards crystal formation was very slow, due to the high viscosity of the glasses.

CORNING GLASS WORKS,
CORNING, N. Y.

N. L. BOWEN: Of the series of glasses mentioned by Mr. Cox, none has a content of barium disilicate as great as that of the corrected light barium mentioned on page 271. It is not surpris-

[1] *Loc. cit.*, p. 277.

[2] *Trans. Am. Ceram. Soc.*, 15, 600 (1913).

ing, therefore, that devitrification was not noted in the barium-rich members of his series. The observance of devitrification in the silica-rich members of the series was probably due to the separation of silica, not barium disilicate.

The simple explanation of surface devitrification offered by Brockbank cannot be accepted as more than a possible contributory circumstance in some glasses. I have frequently observed that devitrification begins at the surface of glasses entirely free from alkalis or any significantly volatile constituents. A crucible full of a glass containing only CaO, MgO and SiO_2 frequently shows a surface skin of crystals of forsterite, Mg_2SiO_4.

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CERAMIC ABSTRACTS.

Provisional Specifications for Glass Refractories, BY THE REFRACTORIES RESEARCH AND SPECIFICATIONS COMMITTEE OF THE COUNCIL. *J. Soc. Glass Tech.*, **3**, 3–14 (1919). *Tank Blocks:* At the request of the purchaser, the following data shall be supplied: (*a*) Analysis, giving SiO_2, Al_2O_3, Fe_2O_3, TiO_2, CaO, MgO, K_2O and Na_2O, (*b*) fusing point in cones, (*c*) porosity, (*d*) linear expansion or contraction after heating to cone 14, (*e*) crushing strength. Tentative specifications governing tank blocks follow: *Fusing point*, not under cone 30; *texture*, free from ironstone or other segregated impurity, uniformly burnt, as little black core as possible, free from holes and flaws and with true surfaces; *porosity*, shall not exceed 18% for flux-line blocks, 23% for replacement flux-line blocks, 25% for bottom-side blocks and 30% for tank-bottom blocks, the porosity being expressed as vol. of pores × 100 ÷ vol. of piece (including pores); *after-contraction* or *expansion*, test pieces about 3 × 1 to 2 × 1 to 2″ heated to cone 14 shall, when cold, show not over 2% expansion or contraction; *crushing strength*, a 4″ cube shall show at least 1,600 lbs. sq. in. *Silica bricks:* At the purchaser's request he shall be given the following: (*a*) Analysis, (*b*) fusing point, (*c*) linear expansion after heating to cone 12. Tentative specifications follow: *Analysis*, SiO_2 not under 94%, CaO not over 2%; *fusing point*, not under cone 32; *after-expansion*, not over 0.75% linear expansion after heating to cone 12; *texture*, uniform burning and absence of holes or flaws; *variations from measurements*, not over ±2%. *Silica Cement:* Shall be finely ground, contain not under 92% SiO_2 and fuse not under cone 32. *Raw Clays and Grog* are briefly covered. While manufacturers are free to use any materials and process that will give products complying with the specifications the suggestions are made as to materials and mixtures. The clay aggregate for tank blocks should usually consist of three portions: (1) *Bind clay* (a fat, aluminous clay); (2) base clay (a strong refractory clay); (3) grog (which should consist of the bind and base clays mixed together in the finely ground state, tempered, and burned to cone 14). The following compositions (after burning) and properties are suggested as indicating suitable clays. *Bind Clay:* Free silica, small proportions only; alumina, 36 to 40%; total alkalies, less than 2.5%; total fluxes, less than 4.5%. It should have a low sintering temperature but should not fuse below cone 31. *Base Clay:* Alumina, 25 to 32%; total alkalies, less than 1.4%; total fluxes, less than 4.0%; fusing point not under cone 31. The following notes are suggestions as to suitable mixtures and methods for the consideration of tank-block manufacturers: (*A*) *Tank Bottoms.* Fat plastic clay, 1 volume; lean or strong clay, 3 volumes. These clays should be well mixed in the dry, ground condition. Grog should be made by tempering a portion of the

above mixture, making it into slabs and burning to cone 10. *Mixture for blocks:* Dry, ground clay of above mixture, 5 volumes; grog $^1/_4$ to $^1/_8$" mesh, 1 volume; grog $^1/_8$ to $^1/_{16}$" mesh, 1 volume; grog $^1/_{16}$ to $^1/_{32}$" mesh, 1 volume. The mix should age for one month at least. Blocks should be slowly dried and burned to cone 10. (*B*) *Bottom-side Blocks.* Fat plastic clay, 2 volumes; lean or strong clay, 2 volumes. Grog should be made from above mixture (as for bottom blocks) and burned to cone 12. *Mixture for Blocks:* Dry ground clay of the above mixture, 5 volumes; grog $^1/_8$ to $^1/_4$" mesh, 1 vol.; grog $^1/_8$ to $^1/_{16}$" mesh, 2 vols.; grog $^1/_{16}$ to $^1/_{32}$", 1 vol. Blocks should be burned to cone 12. (*C*) *Top Side* (*Flux-line*) *Blocks.* Fat plastic clay, 2 vols.; strong clay, 1 vol.; grog should be made by burning the above mixture to cone 14. *Mixture for Blocks:* Dry ground clay of above mixture, 5 vols.; grog $^1/_8$ to $^1/_{10}$" mesh, 1 vol.; grog $^1/_{10}$ to $^1/_{32}$" mesh, 2 vols.; blocks should be burned to cone 14. (*D*) *Replacement* (*Flux-line*) *Blocks:* Fat plastic clay, 1 vol.; lower grade fat clay, 1 vol.; strong clay, 1 vol. Grog should be made by burning the above mixture to cones 6–8. *Mixture for Blocks:* Dry ground clay of above mixture, 5 vols.; grog $^1/_4$ to $^1/_8$" mesh, 2 vols.; grog $^1/_8$ to $^1/_{16}$" mesh, 2 vols.; grog $^1/_{16}$ to $^1/_{32}$" mesh, 1 vol. Blocks should be burned to cones 6–8. To avoid black-coring, the Committee recommends the use of tank blocks with no face having an area greater than 4 sq. ft. Clays suitable for pots may be classified in three grades. The lowest, or Grade 3, corresponds with the material in general use. Grades 2 and 1 are purer and more refractory. The following are limits for the composition (burned samples) and fusion temperature suggested for each grade. *Grade 1—Bind Clay.* Chemical analysis: SiO_2, 60 or less; Al_2O_3, 34 or more; Fe_2O_3, 1.8 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 1 or less. Fusion point, not under cone 32. Mechanical Division. Clay which can be blunged and sieved to pass 80-mesh with not more than 10% residue. *Base Clay.* Chemical Analysis: SiO_2, 55 or less; Al_2O_3, 40 or more; Fe_2O_3, 1 or less; Na_2O and K_2O, 1.5 or less; MgO and CaO, 0.5 or less. Fusion point, not under cone 35. Mechanical Division. Ground to pass 80-mesh. *Grog.* Chemical Analysis: SiO_2, 50 or less; Al_2O_3, 40 or more; Fe_2O_3, 1 or less; Na_2O and K_2O, 1.5 or less; MgO and CaO, 0.5 or less. Fusion point, not under cone 35. Mechanical Division. Ground to pass 12-mesh. Temperature of firing, not lower than cone 14. *Grade 2. Bind Clay.* Chemical Analysis: SiO_2, 65 or less; Al_2O_3, 30 or less; Fe_2O_3, 2 or less; Na_2O and K_2O, 2 or less; MgO and CaO, 1 or less. Fusion point, not under cone 30. Mechanical Division. Clay which can be blunged and sieved to pass through 50-mesh with less than 10% residue. *Base Clay.* Chemical Analysis: SiO_2, 60 or less; Al_2O_3, 35 or more; Fe_2O_3, 2 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 0.6 or less. Fusion point, not under cone 32. Mechanical Division. Ground to pass 50-mesh. *Grog.* Chemical Analysis: SiO_2, 60 or less; Al_2O_3, 35 or more; Fe_2O_3, 2 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 0.6 or less. Fusion point, not under cone 32. Temperature of firing, not lower than cone 12. *Grade 3. Bind Clay.* Chemical Analysis: SiO_2, 70 or less; Al_2O_3, 25 or more; Fe_2O_3,

2.5 or less; Na_2O and K_2O, 2.5 or less; MgO and CaO, 1 or less. Fusion point, not under cone 28–29. Mechanical Division. Ground to 12-mesh. *Base Clay.* Chemical Analysis: SiO_2, 70 or less; Al_2O_3, 25 or more; Fe_2O_3, 2 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 1.0 or less. Fusion point not under cone 30–31. Mechanical Division. Ground to 12-mesh. *Grog.* Chemical analysis: SiO_2, 70 or less; Al_2O_3, 25 or more; Fe_2O_3, 2 or less; Na_2O and K_2O, 1.8 or less; MgO and CaO, 1.0 or less. Fusion point, not under cone 31. Mechanical Division. Ground to pass 12-mesh. Temperature of firing not lower than cone 9. C. H. KERR.

Bottle-glass and glass-bottle manufacture. W. E. S. TURNER. *Jour. Soc. Glass Tech.*, **3**, 37–49 (1919).—Data presented in England has been largely in reference to chemical and scientific glasswares only, but the same principles hold in their application to furnace management, annealing, etc., in the bottle industry. Syphon bottles of English make are now demanded. Analysis of a typical syphon bottle glass from Bohemia follows: SiO_2, 75.34; Al_2O_3, 0.45; Fe_2O_3, 0.30; CaO, 6.83; MgO, 0.21; Na_2O, 13.78; K_2O, 3.41; Sb_2O_3, trace. This corresponds to the batch: Sand, 75.5 parts; limestone, 12.5 parts; soda ash, 27.6 parts; antimony oxide, 0.5 part; manganese (as needed). Another batch recommended for use in Germany follows: Sand, 100 parts; soda ash, 33 parts; limestone, 22 parts; saltpetre, 3 parts; saltcake, 1 part; manganese, 0.3 part; antimony oxide, 0.3 part; cobalt oxide, 0.0005 part. A similar batch recommended by the author for use in England is: Sand, 100 parts; soda ash, 33 parts; limestone, 18 parts; soda niter, 4 parts; arsenic, 0.2 part, manganese, 0.2 part. In most bottle plants the tendency to overcome troubles by liberal increases in alkali is dangerous for durability of the glass is apt to suffer. Bottles for medical and pharmaceutical preparations, also milk bottles, feeding bottles and white flint bottles generally should be tested for durability. Simple tests are (1) 1–2% morphine hydrochloride should be colorless or at most pale yellow after 24 hrs. standing; (2) 0.5–1% strychnine nitrate should show no crystals separating out in 24 hrs.; (3) 1% mercuric chloride should show no precipitation of colored oxide in 24 hrs.; (4) water with a few drops of phenolphthalein in a corked bottle should not develop pink color in 24 hrs. A better test than the above is the autoclave test carried out for 2 hrs. at 120° C. With a simple soda-lime glass the % soda ash in the batch should not exceed 28%. Of the three following batches, No. 1 yields glass that is not durable; No. 2 fairly satisfactory; No. 3 is best: Glass batch No. 1: Sand, 63.19; limestone, 7.02; soda ash, 29.78. No. 2, respectively: 63.26, 8.79, 27.94. No. 3, respectively: 62.86, 15.72, 21.42%. Production in a bottle plant hinges quite largely on the composition of the glass. General limits for easy working glasses are: Soda-lime glasses, 14% CaO; soda-magnesia glasses, 9% MgO; soda-alumina glasses, 9% Al_2O_3, these figures being percentages of the finished glass. These amounts may be increased by (1) adding other oxides; (2) reducing SiO_2. The effects of various constituents on viscosity is shown in the following summary:

Soda (*a*) makes glass more fluid, (*b*) reduces rate of setting; potash resembles soda, but is less effective weight for weight; lime (*a*) makes glass less fluid at low temperatures, but more fluid at high; (*b*) increases rate of setting; magnesia (*a*) makes glass less fluid, (*b*) increases rate of setting, but not to the same extent as lime; alumina (*a*) makes softening temperature high, (*b*) viscosity at high temperature not high compared with lime or silica; silica, causes high viscosity which does not change rapidly. A quick setting German bottle batch for use with the Owen's machine is: Sand, 1000; limestone, 554; saltcake, 300; fluorspar, 60; powdered coal, 24; manganese, 42; iron oxide, 13.

C. H. KERR.

"Silica products." M. A. BIGOT. *La Ceramique*, [1] **21**, 61 (1919).—The raw materials studied were quartzite, flint rock and sandstone. These are heated to different temps. up to 1710° C and the following properties determined: Fusibility, linear expansion, apparent density, sp. gr. by method of Le Chatelier and Candlot, absorption, microscopic examination. Fig. 1

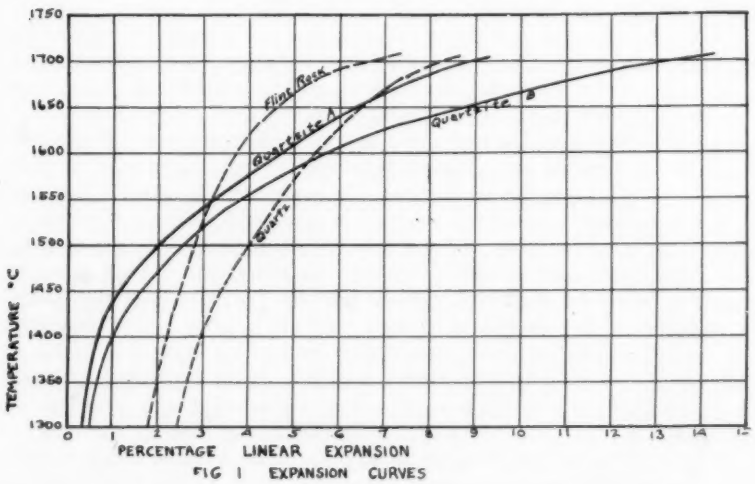


FIG 1 EXPANSION CURVES

shows the relation between linear expansion and temperature for a few silicious raw materials. Fig. 2 shows the relation between absorption and temperature. The finished products studied include those employed in the Martin or similar furnaces. These products are made of sandstone to which 2 per cent lime is added. The finished products are subjected to the above-mentioned tests and to those given in Table 1, which gives results for six commercial brick. In addition to these tests the crushing strength at different temperatures up to 1500° C were determined upon 7 different specimens of refractories by means of a special furnace constructed by Capt. Bodin. Cubes

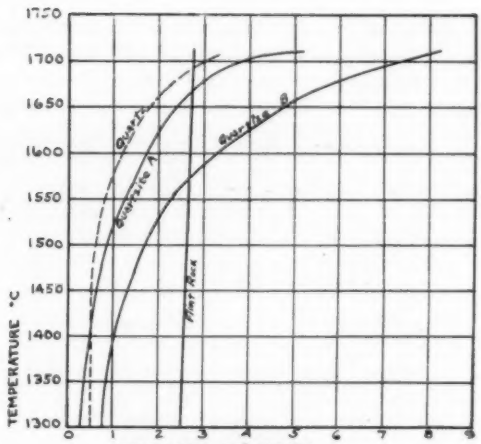


FIG 2. ABSORPTION CURVES

TABLE 1.

Designating letter	A	B	C	D	E	F
Fusion temp.	1750° C	1730	1730	1730	1710	1730
Linear expansion at 1710° C.	1.60%	5.90	4.00	2.50	..	..
Absorption	12.0%	9.5	10.0	10.5	2.1	0.3
Absorption after burning to 1710° C.	9.7%	16.1	12.0	9.0	..	..
Crushing strength, kg. per sq. cm. at ordinary temperatures.	121	480	160	166	1115	1415
Crushing strength, kg. per sq. cm. after burning to 1710° C.	....	0	52	..	..	..
Crushing strength, kg. per sq. cm. at 1500° C after burning to 1710° C.	77	0	0	37	52	138

A. Good commercial brick.
B. Brick which cracked at high temp.
C. Brick made of quartz rock and friable at high temperatures.
D. Brick from Martin furnace, part not attacked.
E. Brick from Martin furnace, brown part.
F. Brick from Martin furnace, gray part.

20 mm. on a side are burned to the desired temp. in a small gas-fired furnace, placed between two cylinders of refractory material and the load applied, a registering device recording the crushing load. The results are shown diagrammatically in Figs. 3 and 4 (Fig. 3 combines Figs. 8, 9, 10 and 11 in the original and Fig. 4 includes Figs. 12, 13 and 14). The reason for the high strength

of some of the refractories in the neighborhood of 1000° C cannot be stated with certainty. Edwards and Rigby[1] determined the coefficient of expansion of a refractory brick between 15° and 1180° C as 81 × 10^{-7}, and between 15° and 1180° C as 61 × 10^{-7}. This may indicate that in the neighborhood of

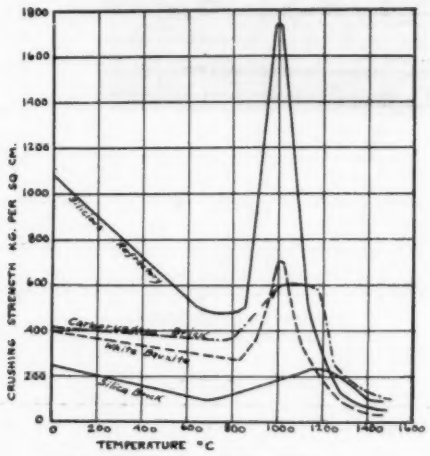


FIG 3 CRUSHING STRENGTH CURVES

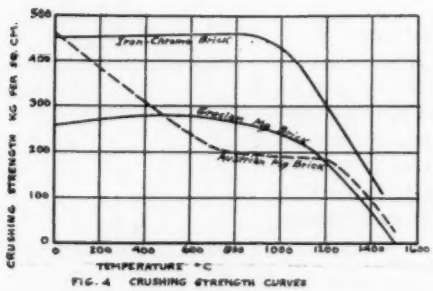


FIG. 4 CRUSHING STRENGTH CURVES

1000° C certain refractories undergo some kind of contraction which causes greater strength. If this explanation is correct, products such as magnesite and chrome brick which do not increase in strength near 1000° C have no such contraction near this temperature. F. A. KIRKPATRICK.

Summary of discussion on the setting of cements and plasters. C. H. DESCH. *Trans. Faraday Soc.*, **14**, 67 (1919).—The question as to whether the setting of a cement itself is a colloidal or a crystalline process has been studied almost exclusively in the United States and France. As regards

[1] *Trans. Eng. Ceram. Soc.*, **16**, 271 (1916–17).

the main point at issue between the supporters of the colloidal hypothesis and Le Chatelier, it is very clearly established that we are dealing largely with a difference of terms; that whether one regards the jelly as a mass of extremely minute interlacing particles or not is not of very much importance. At any rate, the essential point is that in the colloidal substance, the particles are extremely small, and therefore the surface forces are very important. With crystals of perceptible size, such as occur in the crystallization of Na_2SO_4, the surface forces are very small compared to the forces of cohesion. In the case of the ultra-microscopic particles in the colloid, the surface forces are large in proportion to the forces of cohesion. When this fact is thoroughly understood it is seen that there is no great question of fact at issue between the two views. In the setting of metals, on Rosenhain's hypothesis, we are dealing with particles very close together becoming united by exceedingly thin films. These films in the case of cements are often of greater dimensions than the particles from which they start, so that we have a mass of considerable thickness and not a mere intercrystalline film. The essential difference between cases of recrystallization and of the setting of a colloidal mass lies in the solubility. The substances which occur in cements after setting are of very low solubility. It is quite possible that the original substance present is clinker of comparatively high solubility, but that the solution formed by it is unstable and the stable products finally obtained are of extremely low solubility. F. A. KIRKPATRICK.

The mechanism of the setting process in plaster and cement. CECIL H. DESCH. *Trans. Faraday Soc.*, **14**, 1 (1919).—That part of the paper dealing with plaster of Paris and lime mortar is omitted as being already common knowledge. There are two conflicting hypotheses in regard to the setting process of Portland cement. One is the crystallization theory of Le Chatelier put forward in 1887 (see Ceramic Abstracts, THIS JOURNAL, June 1919, for a recent discussion by Le Chatelier). The alternative hypothesis was proposed by W. Michaelis in 1893.[1] On this view, while the chemical reactions assumed are those which were shown to take place by Le Chatelier, the physical conditions are supposed to be different. The hydrated metasilicate is considered to form, not a mass of radiating crystals, but a gelatinous mass or gel, the gradual dehydration of which brings about the hardening of the cement with time. The aluminate crystallizes much more readily than the silicate; but even this is regarded as forming a gel when the solution is sufficiently supersaturated, as is the case in the practical use of cement. The presence of gelatinous material has been shown by a number of workers.[2] An attempt to distinguish between the various possible colloidal products

[1] *Chem. Ztg.*, **17**, 982 (1893).

[2] Stern, *Z. anorg. Chem.*, **63**, 160 (1909); H. Ambrown, *Tonind. Ztg.*, **33**, 270 (1909); Colony, *School Mines Quart.*, **36**, 1 (1914); M. von Glasenapp, *Chem. Ztg.*, **38**, 588 (1914).

has been made by Keisermann,[1] using the method of staining with organic dyes. Others who have used this method are Wetzel[2] and Blumenthal.[3] The staining results depend largely on the concentrations and size of grain of the substances concerned and different workers find difficulty in checking each others' results. The U. S. Bureau of Standards (Tech Papers Nos. 43 and 78) has successfully used the staining method and has been able thus to identify the various gelatinous materials. The real nature of the colloidal condition has been unnecessarily obscured by the barbarous terminology which has been adopted by so many writers on the chemistry of colloids, and especially by the school of Wo. Ostwald. Stripped of these uncouth technical terms, the study of colloids appears far less formidable and it is hoped that the putting in relief of the view of the two rival hypotheses as to the setting and hardening of cement may lead to a recognition of the true facts of the case, and to an elimination from the subject of the complications which owe their origin largely to a defective terminology. F. A. KIRKPATRICK.

Discussion on the setting of cements and plasters, WALTER ROSENHAIN. *Trans. Faraday Soc.*, **14**, 52 (1919).—The setting of cement appears to be one of a whole class of phenomena of an analogous character, and this analogy may be extended still further to include the setting of metals when they solidify from fusion. The latter process is like the former because we see in it the formation of an aggregate of coherent crystals instead of merely a heap of loose crystals lying one upon another, and an aggregate of crystals adhering powerfully to one another is very much what we have in a plaster or cement after setting. We can dismiss at once as the cause of cohesion the idea of mere frictional adhesion due to any sort of interlocking. We have cohesion due to that cause in textile fibres where the frictional adhesion between intertwined units may become very large compared with the strength of any one unit. The hypothesis now generally accepted as to the cause of the adhesion of crystals in metals is that a thin film of amorphous material acts as a binder between the crystals. We may regard cement as consisting, at one stage, of solid grains or crystals lying surrounded by a liquid solution of certain constituents. This liquid then undergoes solidification as the result of the removal of water—either by percolation or by chemical action, such as the hydration of some of the solid material. If the film of liquid is thick enough, this solidification may take place by crystallization, but if it is very thin it may—by analogy with the phenomena in metals—congeal in an amorphous condition. Even if there is crystallization, the final thin films of liquid between the crystals will still solidify as "undercooled," or supersaturated liquid. It is these amorphous layers which give to the whole mass its great stiffness and strength. This theory, which has proved useful in explaining

[1] "Der Portlandzement, sieve Hydratbildung and Konstitution," Dresden (1910).

[2] *Zentr. Hydrauk. Zemente*, **2**, 34 (1911).

[3] Dissert., Jena, 1912; *Silikat Z.*, **2**, 43 (1919).

the setting of metals, should aid in the analogous phenomena met with in the setting of cement. F. A. KIRKPATRICK.

Discussion on setting of cements and plasters. E. HATSCHEK. *Trans. Faraday Soc.*, **14**, 53 (1919).—The reason for the different results obtained by Le Chatelier and Michaelis in regard to the setting of cement is that one worked in dilute and the other in concentrated solution, and this is practically the whole difference, and accounts for their divergent results and conclusions It is well known, especially from the work of von Weimarn on about 200 different substances, that slightly soluble compounds may be obtained either as gels, as the familiar crystalline precipitation, or as sols, simply by an appropriate choice of the concentrations of the reacting solutions. Thus, $BaSO_4$ may be obtained as a clear gel by using concentrated solutions of two exceptionally soluble salts, namely BaSCN and $MnSO_4$. The two theories are not only compatible but even complementary. It is perfectly possible that the first product of a reaction may be a gel consisting of ultra-microcrystalline elements and that this may gradually assume a coarser crystalline structure owing to the intermediate true solution of the reaction product. One case has been described by W. Doehle,[1] who obtained the Hg salt of a sulphonic acid which formed gels in concentrations as low as 1 : 300. The gel gradually breaks up and the salt crystallizes out, the process being irreversible. Another, even more striking case, is that of camphoryl phenyl thio-semicarbazide studied by M. O. Forster.[2] This substance has a true solubility of about 0.5 per cent in cold abs. alc., but is very sol. in boiling alcohol. If the latter solution is allowed to cool slowly, the substance crystallizes out in large, simple crystals. If, however, the solution is cooled suddenly, it sets to a beautiful gel, recalling silicic acid gel. This gel is not stable but in a short time—hours, or at most, days, depending on temperature and concentration—the gel exudes liquid, while at the same time the substance crystallizes out. The crystals thus formed are all twins or more complicated aggregates. This is an instance in which gel formation and subsequent crystallization succeed each other with striking rapidity, owing to the high true solubility of the substance in the cold solvent. Intermediate cases between this and the transformations requiring years or possibly centuries, can, no doubt, be found by a careful study of solubilities.

F. A. KIRKPATRICK.

CERAMIC PATENTS.

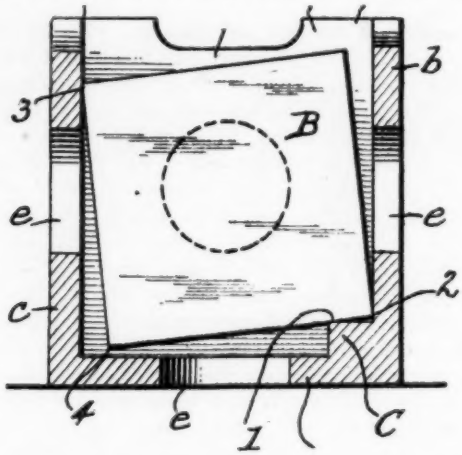
GEORGE E. MIDDLETON, *Abstractor.*

Sagger. H. D. LILLIBRIDGE. U. S. 1,302,684, May 6, 1919. This invention relates to a saggar used in burning floor or wall tiles. Because of the

[1] Ueber Derivate des Benzothiazols, Dissertation, Leipzig, pp. 14, 61 (1912).

[2] *Kolloid Z.*, **11**, 158 (1912).

longitudinal shoulder C at one side of the base of the saggar, the tiles are supported from three points, 1, 2 and 3, the lower corner 4 being unsupported



as shown (see Fig.). The frictional resistance offered by the three points of support prevents displacement of the tiles.

Brick-cutting machine. G. F. PEMBERTON. U. S. 1,302,964. May 6, 1919. A machine for cutting freshly-made, blank, plastic blocks into irregular shapes for use in arches and elsewhere where tapering or trapezoidal shapes are required. The cutter comprises a frame across which wires are strung at the desired angles. The blank is placed on a base plate and this cutting frame forced down through it.

Glass and method of making the same. B. F. DRAKENFELD, JR. U. S. 1,303,268, May 13, 1919. Amblygonite, a lithium-aluminium-fluophosphate of a chemical formula approximating $Li(AL,F)PO_4$ is added to a glass batch, the amblygonite being in a fused condition. When working with a basic batch to be used in the manufacture of jars, bottles, window, plate or other inexpensive glass, from 1 to 5% is added. When making opaque or translucent glass the addition of amblygonite is up to 50% of the weight of the batch. The fluorine and phosphate contents assist in imparting opacity. In all cases the strength, temper and appearance of the glass are said to be improved. Vitreous enamels and glazes are included as well as glasses.

Glass and method of making the same. B. F. DRAKENFELD, JR. U. S. 1,303,269, May 13, 1919. Spodumene, a lithium-aluminium-metasilicate of a chemical formula approximating $LiAl(SiO_3)$ is added to a glass batch. It is said to reduce the coefficient of expansion of the glass. The properties of the resulting glass and the methods of procedure are similar to those discussed under patent 1,303,268, above.

Brick machine. H. R. McCune. U. S. 1,303,344, May 13, 1919. This invention relates to means for roughening and ornamenting the ends and one face of stiff-mud bricks. As the clay ribbon issues from the die of the brick-machine, it is first passed between two brushes rotating vertically in a direction counter to the motion of the clay, these brushes roughening the sides of the clay ribbon, and then under a third brush horizontally rotating in a direction counter to that of the clay, this brush roughening the top of the ribbon.

Brick conveyor. B. S. Moore. U. S. 1,303,359, May 13, 1919. A mechanism for conveying and handling brick, especially adapted for the receiving of the bricks as they are delivered from the molding machine, carrying the same in bulk to another point and depositing them on cars in suitably arranged layers for future handling.

Process of and apparatus for drawing glass. H. T. Bellamy and J. C. Smith. U. S. 1,303,452, May 13, 1919. This invention relates to the drawing of thin-walled glass articles, the specific article illustrated being a cylindrical bulb for small incandescent lamps.

Clay-mixing machine. H. R. Straight. U. S. 1,303,611, May 13, 1919. The mixer consists of an endless conveyor, curved upward to form a trough. Power is applied so that the lower run is taut and the upper being very loose and supported upon a plurality of transverse rollers, presents an undulating surface. Crushed clay is fed onto this trough-shaped conveyor at one end, together with the required water. The mixture then passes under a horizontally rotating cylinder having outstanding pins which act as mixers and then along the undulating surface of the conveyor to the discharge end.

Continuous kiln. D. R. Bone. U. S. 1,303,760, May 13, 1919. This is a continuous kiln of the ring type employing temporary combustible partitions between the green and preheating ware. Separate, auxiliary furnaces are provided for water smoking. Powdered coal for the burn is fed through the platting by means of a rotary feeder which is designed to exclude the entry of air. The inventor seeks to avoid the admission of more air than is necessary completely to burn the fuel used.

Refractory. F. J. Tone. U. S. 1,303,993, May 20, 1919. This invention relates to refractory articles such as crucibles. The composition consists of graphite 40 parts, sintered magnesia 40 parts, plastic clay 20 parts. Such a mixture is said to be easy to mold and work in the plastic state and after burning has a body which is very refractory, less oxidizable than graphite, of superior strength, of high conductivity and great durability.

Manufacture of Keene's cement. J. C. Best. U. S. 1,304,148, May 20, 1919. A process of manufacturing Keene's cement which consists in burning gypsum in a rotary internally-fired kiln for a period of substantially one hour at a temperature of from 1200° to 1400° F., admixing with the burnt product one per cent of a neutral catalyst such as K_2SO_4 and grinding to 150 mesh. This cement is particularly adapted for making artificial marble or scagliola.

Shaft kiln. E. SCHAMATOLLA. U. S. 1,304,514, May 20, 1919. A vertical shaft-kiln for burning lime, magnesite and other rock products with producer gas. The object is to prevent overburning of the product near the lining and gas inlets and underburning near the center, or down-rolling of unburnt rock through the center. This is accomplished, first, by the use of two series of draw openings, the one for the material near the wall and the other for that in the center, by means of which the product near the walls can be drawn oftener than that in the center, and second, by allowing a part of the air needed for combustion to enter at the base of the column whereby complete combustion and overburning near the gas inlets is obviated.

Copies of the above patents may be obtained for five cents each by addressing the "Commissioner of Patents, Washington, D. C.

ACTIVITIES OF THE SOCIETY.

The following motions have recently been passed by the Board of Trustees:

July 8, 1919. It was voted that all papers presented before the Sections and Divisions of the Society be subject to prior publication in the Journal, and that this be incorporated in the Rules of the Society.

July 15, 1919. It was voted to loan a set of the Transactions of the Society, including the JOURNAL, to the Division of Chemistry and Chemical Technology of the National Research Council, as part of a technical library.

July 15, 1919. Credentials were issued to Dr. E. W. Washburn as a delegate to the International Chemical Council, called together in London, for the purpose of completing the organization of an International Chemical Union.

NECROLOGY.

Clarence W. Hathaway, metallurgist for the past eight years for the Granite City Steel Works Branch of the National Enameling and Stamping Co., met his death by drowning while swimming with a party of friends in the Mississippi River, on July 19, 1919, at Granite City, Illinois.

He was born in Columbus, Ohio, in 1886, and was educated in the schools of that place and at the Ohio State University. He had previouly been employed with the U. S. Steel Corporation at Gary, Indiana, before accepting his last position. He was a man of exceptional ability, a keen investigator and a tireless worker.

Mr. Hathaway was a member of the Masons and Elks, American Chemical Society, Metallurgical Society of America, and the American Ceramic Society. He leaves a wife, father and mother, one brother and four sisters.

$6,831.35

The Value of Fuel Saved in One Year

¶ Actual records were kept for 365 days of the burning of 9-inch, high-grade, refractory brick in one continuous tunnel kiln and in seven 30-foot round kilns, during which time 5,110,000 brick were burned in the tunnel kiln as against 5,040,000 in the seven round kilns.

¶ But this isn't all—the actual labor saving amounted to $5,808.00. Taking into consideration the necessary items of depreciation, interest on plant, maintenance and repairs, the average yearly cost for burning 1,000 brick in the continuous tunnel kiln system was $2.95 as against $6.20 in the round kilns.

¶ If you are really interested in the greater efficiency of burning, you will let us tell you more about the actual accomplishments of

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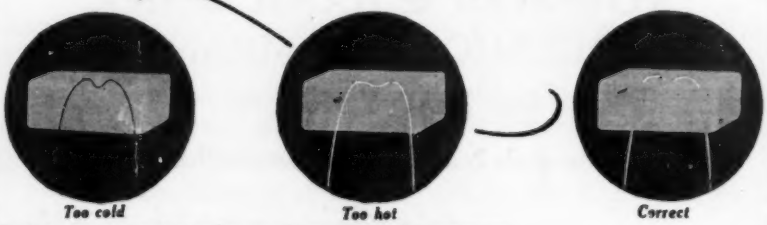

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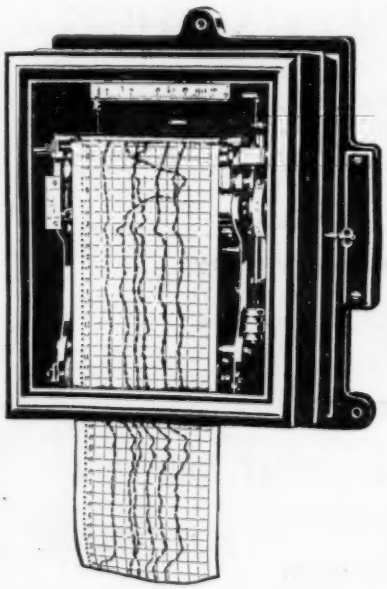

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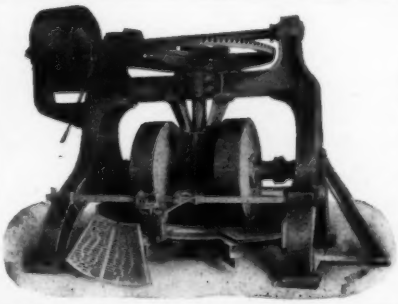

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